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AN INTRODUCTION

TO

PRACTICAL CHEMISTRY

LONDON
URCHILL, NEW BURLINGTON STREET
1878

AN INTRODUCTION

TO

PRACTICAL CHEMISTRY

INCLUDING ANALYSIS

BY

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SEVENTH EDITION

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"Facts are the materials of Science; but all Facts involve Ideas. Since, in observing Facts, we cannot exclude Ideas, we must, for the purposes of Science, take care that the Ideas are clear, and rigorously applied."

Whewell, *Philosophy of the Inductive Sciences*, vol. i. p. xxxvii.

PREFACE

TO THE SEVENTH EDITION.

In this Edition the systematic course of Qualitative Analysis has been arranged in the form of tables, which will be found greatly to assist the student using the work in the laboratory.

Since the publication of the former Edition, the atomic system of notation has been very generally adopted in this country, and it has therefore been deemed advisable to recur to the use of symbols and equations which will be found to be largely used throughout the work. No complete change has been made in the names applied to compounds, since the older names are still often found on the labels in laboratories. The more modern names are, however, introduced occasionally, in order that the student may gradually become accustomed to them. In Part V., devoted to Reagents, where the older names of the tests are arranged alphabetically, the new names have been added, together with the chemical formulæ of the substances.

A Table of Symbols and Atomic Weights of the Elements has been introduced into the Appendix. The Testing Tables in the Appendix have been made to include the rarer metals and acids (inorganic) which are not treated in the systematic course of analysis.

KING'S COLLEGE, LONDON,
April, 1878.

PREFACE

TO THE FIRST EDITION.

Among the many recent and valuable works on Chemistry, I am not aware of one having for its special object to explain and render simple to the beginner, the various processes employed in analysis, or which have been devised for the illustration of the principles of the Science. Most of them contain much that is superfluous for the general student, who has but a limited time to devote to the subject; while they are wanting in those explanatory details, without which he must often fail to understand the rationale of the operations through which he is conducted.

It is with a wish to supply this deficiency, and at the same time to furnish a text-book for my own classes, that the present little work has been written; and as it is intended for the use of those who have made but little progress in the Science, my endeavour has been, throughout, to make everything as simple and intelligible as possible. The employment of complicated or expensive apparatus has been almost wholly avoided.

The outline of most of the First Part was arranged some years ago by my friend Professor Miller (at that time Demonstrator of Chemistry in King's College), for the use and direction of the class of Chemical Manipulation, then first established to supply a growing demand, and to meet the requirements of the University of London, and some of the other examining Boards of the Metropolis. In the compilation of the Second and Third Parts, I have been much indebted to the excellent works of Rose, Fresenius, Parnell, and others; I must also here thank my colleague, whose name I have already mentioned, for many valuable suggestions, and for his kindness in revising the proof-sheets, without which assistance many errors would have crept in, and rendered the book less worthy of the student's confidence.

JOHN E. BOWMAN.

KING'S COLLEGE, LONDON,
September, 1848.

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PART I.

CHAPTER I.*

INTRODUCTORY—GENERAL RULES.

1. So essentially is chemistry an experimental science, and so almost exclusively is it built up of facts which have been elucidated by experiment, that without experimental illustrations it would be quite impossible to teach or to study it with any great amount of success. It is not enough, however, for the student to see experiments performed by others; he must, if he would master even the general principles of chemistry, learn to make experiments himself; and he will, probably, be surprised how much more easily he will retain in his recollection those phenomena (as well as the principles they illustrate) which his own hands have been the means of producing. This is especially the case when he is enabled, while operating in the laboratory, to learn and study the theory of the changes which take place under his direction.

2. It is very important that the student should at once begin to make careful notes of all the experiments he engages in. He should endeavour to do this in as concise and methodical a manner as possible, and he will find it very advantageous to make use of symbols in describing the substances he employs, and the changes which they undergo: he will thus be able to record much in a small

* The beginner is advised to study the Glossary of Chemical Terms in the Appendix.

space, and at the same time he will be making himself familiar with the composition of the substances with which he is experimenting.

3. When, as is often the case, especially in analytical experiments, there are several solutions and precipitates either filtering, digesting, or waiting till the operator has leisure to attend to them, it is necessary to mark them in some way, to prevent confusion. This is easily done with small pieces of gummed paper, on which a letter or number may be written, corresponding with a similar reference mark in the note-book.

4. The student will soon learn by experience that he cannot be too methodical in his operations, or too careful in cultivating habits of neatness and cleanliness. The presence of a little saline or other impurity in a glass, owing to careless washing, or a little extraneous matter having been allowed to find its way into a bottle or test-tube, may retard or spoil the result of whole days of labour.

5. It is easy to clean even the dirtiest vessel, provided it has not been allowed to remain long with the impurities adhering to it; this, indeed, should never be permitted, and is readily avoided by making it a rule never to leave work for the day until the whole of the soiled apparatus has been thoroughly washed and left to drain during the night, ready for wiping the next morning. For most purposes of cleaning, water will be found sufficient, especially when the dirt is still moist; and when mere rinsing does not remove it, gentle friction with moist tow and coal ashes or sand will, in most cases, prove effectual. When the form of the vessel to be cleaned is such as will not allow the introduction of the hand (as flasks, test-tubes, &c.), a small bottle-brush, or a piece of stick or wire, having a little tow wrapped round the end, will be found very convenient. Glasses or basins that have been set aside to drain should, before use, be wiped with a dry clean cloth, to remove any adhering particles of dust or moisture. Bottles or flasks, when required to be perfectly dry inside, may, after most of the water has been removed, be easily dried by warming them gently, and drawing air into them through a glass tube, with the mouth; in this way the water is converted into vapour, which is quickly removed by the current of comparatively dry air.

When a glass or dish is greasy, it should be first wiped as clean as possible with tow or a dry cloth, then moistened with a little strong potash, and lastly, well washed and rinsed with water. When the dirt to be removed is resinous also, or tarry, the application of strong potash or sulphuric acid will generally act upon it in such a way, that subsequent washing with water, together with gentle friction with sand, will render it quite clean. It often happens, especially when a glass has been allowed to dry in a dirty state, that an insoluble crust is formed on the surface which is very difficult of removal by mechanical means, but readily yields on the application of a few drops of hydrochloric or some other acid. An instance of this is afforded by solutions of lime, which, on exposure to the air, frequently deposit a crystalline sediment of carbonate of lime, which adheres strongly to the glass, but instantly dissolves on the addition of the acid.

6. When thrown upon his own resources, the student will often find it of the utmost value to be able to substitute, in default of more perfect apparatus, the common things used in domestic life, which are to be found in every house, such as glasses, plates, cups, saucepans, &c. When in addition to these he has at his command a blowpipe, a small piece of platinum foil and wire, a flask or two, a funnel, and a little glass tubing of different sizes, he will, with the exercise of a little ingenuity and contrivance, be able to go through a very considerable course of experimental chemistry. He may rest assured that it is no disadvantage, but rather the contrary, to be thus compelled to devise and construct for himself rude and extemporaneous forms of apparatus; and if he should require encouragement to persevere in spite of the scantiness of his resources, he need only be reminded that the majority of those whose names shine brightest in the annals of science, have laid the groundwork of their future eminence while placed under the most unfavourable circumstances. So it was with the great Davy;* so with Dalton, with Scheele,

* "His means, of course, were very limited; not more extensive than those with which Priestley and Scheele began their labours in the same fruitful field. His apparatus, I believe, consisted chiefly of phials, wine-glasses, and tea-cups, tobacco-pipes, and earthen crucibles; and his materials were chiefly the mineral acids and the alkalies, and some other articles which are in common use in medicine."—*Life of Sir H. Davy*, by John Davy M.D., vol. i. p. 43.

Faraday, Dumas, Liebig, and many others almost equally illustrious.

Memoranda on the use of Symbols and Equations.

Each symbol represents an *atomic weight* of the element, as given in the Table in the Appendix.

The atomic weights are the relative weights of the smallest particles which can enter into combination.

Compounds of two or more elements are represented by their symbols written close together, with small figures annexed to indicate the number of atoms.

Thus, hydrochloric acid, HCl, a compound of one atom of hydrogen with one atom of chlorine.

Sodium phosphate, Na_2HPO_4, composed of two atoms of sodium, one of hydrogen, one of phosphorus, and four of oxygen.

The formula of a compound body always represents a molecular weight of the compound.

The molecular weights are the relative weights of the smallest particles which can exist in a separate form. Thus HCl represents 36·5 parts by weight ($H=1+Cl=35{\cdot}5$).

The substances which are brought together to effect a chemical change are written on the left of the equation, and separated by the sign +; the resulting products are written on the right. Thus, the equation

$$AgNO_3 + HCl = AgCl + HNO_3$$

implies that silver nitrate and hydrochloric acid produce silver chloride and nitric acid.

The learner will find no difficulty in writing a large number of equations if it be remembered that—

1. *The metals usually change places with hydrogen.*
2. *The following metals change places with one atom of hydrogen:* K, Ag, Na.
3. *The following metals change places (usually) with two atoms of hydrogen:* Ba, Ca, Co, Cu, Fe, Pb, Mg, Mn, Ni, Sr, Zn.
4. *The following metals change places (usually) with three atoms of hydrogen:* Al, Sb, As, Bi.

The learner is strongly recommended to exercise himself in explaining every chemical change by an equation.

CHAPTER II.

MANIPULATION WITH GASES.

SECTION I.

Preparation of Hydrogen (II).*

7. Weigh 300 grains of granulated zinc, and introduce the fragments carefully through the tubulure of a retort, sliding them, not dropping them in, to avoid the risk of breaking the bottom of the retort, which is usually of thin glass, and consequently seldom strong enough to bear a blow without injury.

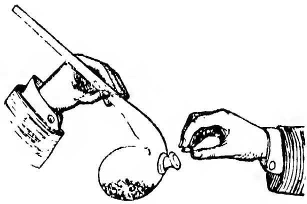


Fig. 1.

Pour upon the metal four fluid ounces of dilute sulphuric acid, consisting of one part by measure of oil of vitriol and five parts of water.

(Oil of vitriol, when mixed with water, evolves much heat; hence they must be mixed in a vessel of earthenware or of *thin* glass, into which the water is poured first, and the sulphuric acid added, in a thin stream, whilst the water is stirred with a glass rod.)

Effervescence immediately commences, owing to the evolution of the gas, the zinc being rapidly dissolved, in the form of sulphate,† with evolution of much heat, and some black flakes of lead being separated, since that metal is nearly always found in commercial zinc, and is not dissolved by dilute sulphuric acid. The gas may be collected in cylinders filled with water, and inverted upon the shelf of the pneumatic trough (fig. 2.)

* The specific gravity, or relative weight, of hydrogen is lower than that of any other form of matter, being only 0·069, that of common air being considered 1·000. 100 cubic inches, at 60° F. and 30 in bar., weigh 2·14 grains.

† $H_2SO_4 + Zn = H_2 + ZnSO_4$.

A three-legged table made of tin-plate, and perforated with two or three holes, may be placed in any deep vessel of water to be used as a pneumatic trough. The so-called *bee-hive shelves* of earthenware are also used for this purpose.

Fill five or six gas-cylinders with hydrogen before commencing the following experiments, and set the retort aside upon a list ring.

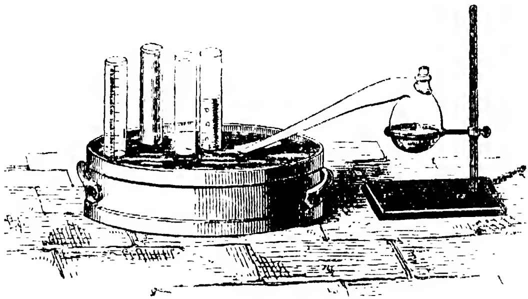


Fig. 2. Pneumatic Trough.

8. Lift the cylinder which was first filled off the shelf, still keeping its mouth downwards, and introduce a lighted taper; a slight explosion will probably ensue, because the hydrogen is mixed with atmospheric air previously contained in the retort; showing that *a light must never be applied to a hydrogen apparatus until all the air has been expelled.*

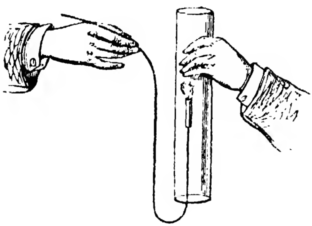


Fig. 3

Repeat the experiment with a cylinder of hydrogen free from air; the taper is extinguished, for the gas does not support combustion, but the hydrogen takes fire at the mouth of the cylinder with a slight report, and if the taper be carefully withdrawn, it may be rekindled at the flame of the hydrogen, and this extinction and rekindling

of the taper may be repeated until the gas has burnt out; H_2+O (from the air)$=H_2O$ (*water*).

9. Fill a small cylinder with the gas, and having removed it from the shelf, let it stand for a few seconds with its open end *upwards*. If a lighted taper be now applied (fig. 6), no combustion will ensue, as the hydrogen will have escaped upwards, on account of its low specific gravity.

10. Repeat the last experiment, holding the cylinder with the open end *downwards*. On applying a lighted taper, a slight explosion will take place, showing that the hydrogen had not entirely escaped as before, but had become mixed with air by *diffusion*.

11. Raise a cylinder of hydrogen from the trough, keeping its mouth downwards, and pour the gas upwards into another cylinder containing air. Prove the transfer of the gas by applying a lighted taper to the mouth of each cylinder.

12. Transfer some of the gas from a large cylinder to a small one, and from this again to tubes, until it can be done without allowing any bubbles to escape.* When the gas is to be decanted into a cylinder or tube which is much narrower, it may be first transferred into a lipped glass; or an inverted funnel may be used, as shown in fig. 4.

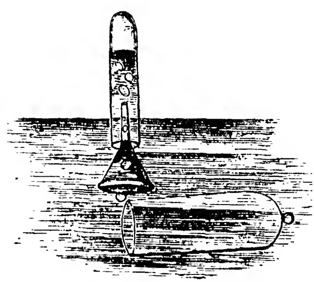


Fig. 4.

13. Transfer a little of the hydrogen in this way into a graduated cylinder; measure out two cubic inches, and transfer them into another cylinder.

The graduated cylinder being filled with water, close its mouth, under the water, with a glass plate, then raise it, with the glass plate beneath, out of the water, and slip the plate gradually aside, so that bubbles of air may enter; measure out, in this way, two cubic inches of air, slide the

* It is advisable to practise this with a jar containing air, in order to avoid wasting the hydrogen.

plate back again so as to close the cylinder, and decant the air, under water, into the cylinder containing the two cubic inches of hydrogen; then close this with a glass plate, place it mouth upwards upon the table, remove the plate, and apply a lighted taper.

Repeat the experiment a second time, with two cubic inches of hydrogen and four cubic inches of air; and a third time, with six cubic inches of air, and ascertain in which case the loudest explosion takes place. Compare the conclusion arrived at with that expected from the

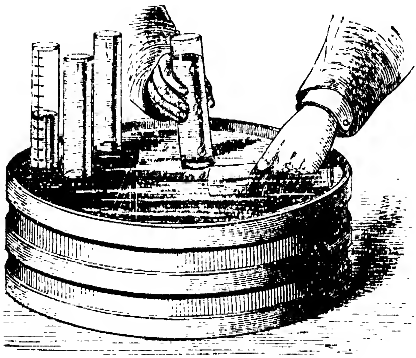


Fig. 5. Decanting Gas

composition of air and the combining volumes of oxygen and hydrogen.

SECTION II.

Preparation of Carbonic Acid Gas (CO_2).*

14. Put 300 grains of marble (carbonate of lime, $CaCO_3$) broken into fragments about the size of a pea, into a retort, observing the same precautions as were recommended in the preparation of hydrogen (7). Measure out an ounce and a half of hydrochloric acid (HCl), dilute it with an equal quantity of water, and pour the mixture

* The specific gravity of carbonic acid is 1·529 (air being 1·0), 100 cubic inches weighing 47·3 grains. At a temperature of 60° water dissolves about its own bulk of carbonic acid.

upon the marble. The gas is immediately given off, causing brisk effervescence, and it may be collected in jars placed on the shelf of the pneumatic trough, the first jar-full being rejected as impure.*

15. Introduce a lighted taper into a small jar of the gas held with its open end upwards. It is instantly extinguished; and as the carbonic acid remains some time in the jar, on account of its high specific gravity, the taper may be extinguished repeatedly in the same jar of gas.

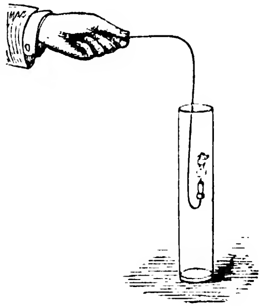


Fig. 6.

16. Pour a little lime-water into a test glass, and thence into a jar filled with the gas,† closing the mouth of the jar with a glass plate, and agitating the gas and liquid together. The lime-water almost immediately becomes milky, owing to the formation of carbonate of lime, which is insoluble in water. Pour a little of this milky fluid into another jar of carbonic acid; on closing with a glass plate and shaking violently, the milkiness will disappear, for the excess of carbonic acid dissolves the carbonate of lime.

If a few drops of hydrochloric acid be added to the other portion, the carbonate of lime is decomposed, and the milkiness disappears, chloride of calcium being formed, which is soluble in water.

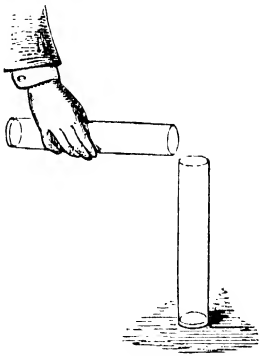


Fig. 7.

17. Having filled a jar with the gas, pour it into another jar somewhat smaller (fig. 7); this is easily effected, owing to the high specific gravity of carbonic acid. Test its presence in both

* $CaCO_3 + 2HCl = CaCl_2 + H_2O + CO_2$.

† If the lime-water were poured from the bottle at once into the gas-jar, a quantity of the carbonic acid would enter the bottle and render the whole of the lime-water turbid.

jars with lime-water, and by its power of extinguishing a taper.

18. Allow a little of the gas to escape, under water, from a jar filled with carbonic acid, and when a little water has thus entered the jar, close it with the palm of the hand, remove it from the trough, and shake violently to and fro; a portion of the carbonic acid will be absorbed by the water, and on plunging the jar again beneath the surface and removing the hand, more water will enter to supply the place of the gas which had been absorbed; on adding some lime-water to the water in the jar, the presence of carbonic acid will be rendered evident.

19. By means of a narrow tube open at both ends, fill a jar over the pneumatic trough, with air from the lungs. Test it with a lighted taper, and observe that it causes an abundant precipitate in lime-water, owing to the presence of carbonic acid.

20. Invert a jar filled with common air over a lighted taper fixed in a cork floating on the water of the pneumatic trough (fig. 8); observe that it soon burns dimly, and is shortly extinguished, the water at the same time slowly rising in the jar. The absorption of air is here owing to the disappearance of part of the atmospheric oxygen, which combines with the hydrogen and carbon of the burning wax, forming water which condenses upon the sides of the jar, and carbonic acid gas, which is equal in volume to the oxygen consumed to form it, and extinguishes the taper long before the whole of the oxygen is exhausted. When the combustion is over, transfer the air into another jar, and test it with lime-water for carbonic acid (16).

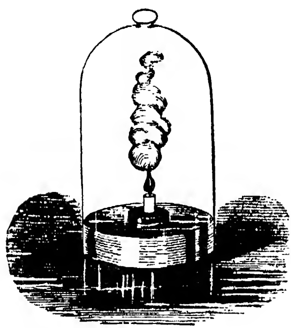


Fig. 8.

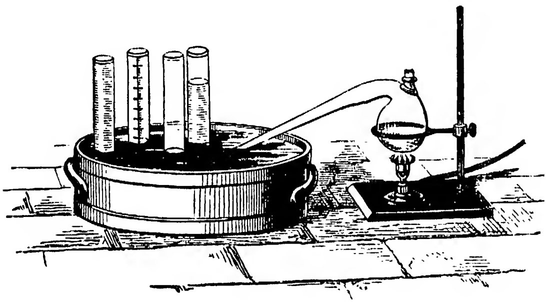
21. Expose a few drops of lime-water to the air upon a clean glass plate, and observe the film of carbonate of lime produced after a few minutes by the atmospheric carbonic acid.

SECTION III.

Preparation of Nitric Oxide (NO.)*

22. Put 300 grains of copper turnings into a retort, and pour upon it three ounces of dilute nitric acid (HNO_3), containing equal measures of the strong acid and water. Decomposition immediately commences on applying a gentle heat (fig. 9), and the nitric oxide is evolved, the copper being dissolved in the form of nitrate of copper, $Cu(NO_3)_2$, which gives the blue colour to the liquid.

$$8HNO_3 + Cu_3 = 3Cu(NO_3)_2 + 2NO + 4H_2O.$$



The gas which is first formed becomes orange, owing to its conversion into nitric peroxide (NO_2) by combining with the atmospheric oxygen contained in the retort.

Before removing the flame from under the retort, be careful to take the neck out of the water, or the latter will be forced up into the retort as it cools, and may either break or overbalance it.

23. Transfer a little to a jar, and test it with a taper;† observe the orange fumes of peroxide of nitrogen, which are instantly produced wherever the gas mixes with the air.

* The specific gravity of nitric oxide is 1·039, 100 cubic inches weighing 32·22 grains.

† Pure nitric oxide extinguishes a taper, but the gas obtained by the above process often contains nitrous oxide which enables it to support combustion.

24. Measure $2\frac{1}{2}$ cubic inches of the gas in a graduated cylinder, and transfer it to another jar over the pneumatic trough: then measure off an equal volume of atmospheric air, and add it, by decantation, to the nitric oxide. When the orange fumes have disappeared, owing to the absorption of the peroxide of nitrogen by the water, transfer it again to the graduated jar, and observe the volume of the mixture, noticing accurately the difference between this and the sum of the original volumes employed before mixing. This experiment should be repeated three or four times, and if the results in each case agree pretty closely, take the average of the experiments, and the amount of condensation, divided by three, should give nearly the quantity of oxygen contained in the atmospheric air employed. Two volumes of nitric oxide when combined with one volume of oxygen form peroxide of nitrogen, which is absorbed by the water; consequently, one-third of the gas absorbed consists of atmospheric oxygen. If the experiment be carefully performed, the absorption will be found to be equal to about three-fifths of the volume of common air employed (*i.e.* to about $1\frac{1}{2}$ cubic inch), the proportion of oxygen contained in it being one-fifth.

Though the results obtained in this way are not very accurate, owing to the formation of other oxides of nitrogen, the experiment is useful as an exercise in the measurement of gases.

SECTION IV.

Preparation of Olefiant Gas (C_2H_4).*

25. Pour into a retort six fluid drachms of alcohol, and add to it in small portions an ounce and a half of strong sulphuric acid, gently agitating the mixture after each addition. Apply heat (fig. 9), and take care that the black froth which is formed towards the close of the operation does not boil over. Collect the gas in jars over the pneumatic trough.†

* The specific gravity of olefiant gas is 0·978.

† $\underset{\text{Alcohol.}}{C_2H_6O} - \underset{\text{Water.}}{H_2O} = C_2H_4.$

$2\,C_2H_6O - H_2O = \underset{\text{Ether.}}{C_4H_{10}O}.$

Observe the odour of ether possessed by the bubbles at the beginning of the process, and that of sulphurous acid towards the end. The olefiant gas has a peculiar odour of garlic.

26. Examine a small jar-full with a taper, and observe that, though the taper is extinguished, the gas burns with a bright white flame, a property which constitutes its great value as the chief illuminating constituent of coal-gas. $C_2H_4+O_6=2CO_2+2H_2O$.

27. When mixed with an equal volume of chlorine, the two gases combine, forming a heavy oily compound, called Dutch liquid. ($C_2H_4Cl_2$.).

The oil collects in drops on the sides of the jar and on the surface of the water, while the gases are gradually absorbed.

Olefiant gas derives its name from the circumstance of its forming this oily compound.

28. Mix together one volume of olefiant gas and two volumes of chlorine; close the jar with a glass plate, and quickly remove it from the pneumatic trough. Apply a light to the mixed gases, and observe the dense cloud of carbonaceous matter that is formed as the combustion gradually passes down the jar, hydrochloric acid being at the same time produced, the chlorine combining only with the hydrogen of the olefiant gas, and liberating the carbon. $C_2H_4+Cl_4=4HCl+C_2$.

SECTION V.

Preparation of Chlorine (Cl.)*

29. Arrange the apparatus as in the preparation of nitric oxide (fig. 9). Introduce into the retort 250 grains of black oxide of manganese (MnO_2) and pour upon it two ounces of common hydrochloric acid (HCl); mix them well by agitation, and apply a very gentle heat. Collect the gas in jars filled with water, taking care not to allow any to escape into the air. The jars will be seen to be filled with the gas by its bright greenish-yellow colour.†

30. Drop a piece of blue litmus paper into one of the

* The specific gravity of chlorine is 2·47.

† $MnO_2+4HCl=MnCl_2+2H_2O+Cl_2$.

jars, when it will be perfectly bleached. Close the jar again with the glass plate to prevent the escape of the irritating chlorine.

31. Immerse a burning taper in a jar of chlorine. The hydrogen in the wax combines with the chlorine and burns with a red flame, giving off fumes of hydrochloric acid mingled with fine particles of carbon.

32. Transfer a little of the gas, under water, from one jar to another, so that some water may enter the first jar. Close this firmly with the palm of the hand, remove it from the water, and shake it violently, when the water will dissolve about twice its volume of chlorine. The suction upon the hand indicates this absorption of the gas; again invert the cylinder under water, and remove the hand, when twice as much water should enter. Test the solution with litmus paper and with nitrate of silver.

33. Mix equal volumes of hydrogen and chlorine over water, place the jar upon the table and apply a lighted taper, when the gases will combine with explosion, forming hydrochloric acid.*

34. Wet a piece of paper with solution of ammonia, and dip it into a jar of chlorine. Thick white clouds of hydrochlorate of ammonia are produced; the chlorine seizing the hydrogen of the ammonia to form hydrochloric acid (which combines with another portion of ammonia), whilst the nitrogen is liberated. If the ammonia be very strong, a pale flash of flame attends the decomposition.

$$4NH_3+Cl_3=3NH_4Cl+N.$$

SECTION VI.

Preparation of Carbonic Oxide (CO).†

35. Carbonic oxide is prepared by the action of strong sulphuric acid on oxalic acid. When a mixture of the two acids is warmed, the oxalic acid is resolved into carbonic acid, carbonic oxide, and water, which latter unites with the sulphuric acid. $H_2C_2O_4-H_2O=CO_2+CO$.

The carbonic oxide is purified from the carbonic acid by passing it through a solution of potash or milk of lime.

* Direct sun-light would also cause the explosion of the mixture.

† The specific gravity of carbonic oxide is ·967.

36. Adapt a cork to a wide-mouthed bottle capable of holding half a pint of water, and fit to it two tubes, one of which, *a* (fig. 10), should be about half an inch in diameter, straight, and sufficiently long to reach nearly to the bottom; the other, *b*, should only just pierce through the cork, and should be bent so as to deliver the gas, as shown in the figure: the diameter of this tube need not be more than about $\frac{1}{3}$ of an inch.

The flask is fitted with a cork, which should be bored to allow the bent tube *c* to pass through it; and care must be taken that this tube is sufficiently small to slide easily

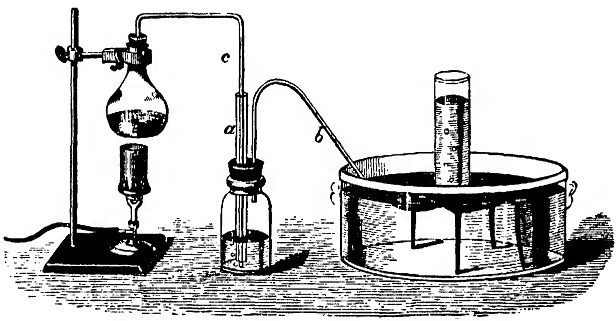


Fig. 10. Preparation of Carbonic Oxide.

down the tube *a*, and long enough to reach to the bottom of the bottle, into which enough water is poured to cover the bottom and the end of the tube *a*.

37. Charge the flask with 180 grains of crystallized oxalic acid and two fluid ounces of strong sulphuric acid. On applying a gentle heat, the gas is given off, the first portions of which must be rejected as impure, and then two or three jars-full may be collected over the pneumatic trough. The gas thus obtained is a mixture of equal volumes of carbonic acid and carbonic oxide.

38. Having collected two or three jars-full of the mixed gases for comparison, withdraw the tube *c* for a moment, and introduce two ounces of a moderately strong solution of potash, through the tube *a*; replace the tube *c*, and proceed to purify the gas from carbonic acid, by passing

it through the alkaline solution in the bottle. Pure carbonic oxide may then be collected.

39. Agitate a little lime-water with a jar-full of the unpurified gas; the presence of carbonic acid is shown by the formation of carbonate of lime.

40. Repeat the experiment with a jar-full of the purified gas. No precipitate ought now to appear.

41. Apply a lighted taper to a jar-full of the impure gas, and observe the characteristic pale-blue flame with which the carbonic oxide burns. $CO+O=CO_2$.

42. Do the same with a jar of the pure gas: the flame is brighter than when carbonic acid was present.

43. Pour a little lime-water into the jar used in the last experiment immediately after the combustion of the gas. The white precipitate which now appears, and which was not formed when the same gas was tested previous to the combustion, shows the result of that process to have been the formation of carbonic acid.

SECTION VII.

Preparation of Oxygen (O).*

44. Weigh 100 grains of chlorate of potash ($KClO_3$) in powder, mix it with 20 grains of well-dried black oxide of manganese,† and place the mixture in a plain retort; adjust the retort so as to deliver the gas under the shelf of the pneumatic trough (fig. 11), and apply the heat of the lamp.

45. The chlorate of potash is thus decomposed, and gradually gives off the whole of its oxygen, while chloride of potassium remains in the retort, together with the oxide of manganese, which is not decomposed during the process.‡

The first portions of the gas should be rejected as im-

* The specific gravity of oxygen is 1·1057, 100 cubic inches weighing 34·20 grains.

† Test a little of the oxide of manganese by mixing *on a piece of paper* with some chlorate and heating on the point of a knife or a piece of glass or porcelain to see that it does not explode. Dangerous accidents have arisen from antimony sulphide having been mistaken for the manganic oxide.

‡ The oxide of manganese is here used, because it is found that, when thus mixed, chlorate of potash gives off its oxygen with much greater facility and at a lower temperature than when heated alone. $KClO_3=KCl+O_3$.

pure, being mixed with the common air contained in the retort.

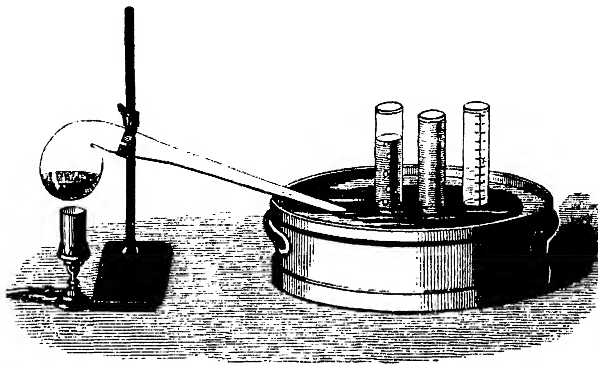


Fig. 11.

46. The jars used for the following experiments should be open both at the top and bottom, the edges of both being ground smooth, so as to be closed air-tight with a glass plate *b* (fig. 12).

Fig. 12.

When filled with gas they are removed from the trough by sliding under them plates or saucers, which will retain enough water to prevent the escape of gas. Stoppered jars are more convenient than those closed with glass-plates.*

47. Fill a jar with the gas, and introduce a glowing taper; it will instantly burst into flame, and burn with great brilliancy, until most of the oxygen is exhausted by combining with the carbon and hydrogen of the wax.

48. Introduce into another jar of the gas a small piece of ignited charcoal, attached to the end of a wire thrust through the cork in the neck of the *deflagrating-collar* (*B*, fig. 13). It bursts into vivid combustion, combining with the oxygen, and forming carbonic acid, the presence

* Stoppered bottles which have been cracked at the bottom are convertible into gas-jars by leading the crack round the bottle with a red hot iron.

of which may be proved by agitating a little lime-water in the jar (16). $C + O_2 = CO_2$.

49. Repeat the experiment with a small coil of thin iron wire, to which a little charcoal or amadou should be attached and ignited, for the purpose of heating the iron sufficiently to cause it to burn. A watch-spring (*A*, fig. 13), deprived of elasticity by being heated in a flame, then made into a coil and filed very bright and thin at one end, which is warmed and dipped into flowers of sulphur, will burn very brilliantly in the jar of oxygen (*C*) if the sulphur be first kindled. The iron combines with the oxygen, forming the black oxide, fused globules of which drop to the bottom, and should be received in water, as they are so intensely hot as to fuse into the glaze of a plate if allowed to fall upon it. $Fe_3 + O_4 = Fe_3O_4$.

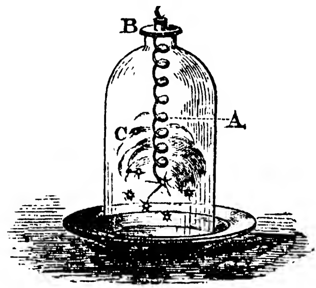


Fig. 13.

50. Place a fragment of sulphur in a deflagrating spoon (*A*, fig. 14), set it on fire by holding it over a lamp, and introduce it into a jar of the gas; the sulphur burns with a brilliant blue flame, combining with the oxygen, and forming sulphurous acid. $S + O_2 = SO_2$.

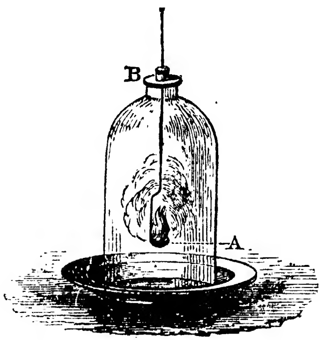


Fig. 14.

51. Repeat the experiment with a very small piece of phosphorus,* previously dried by careful pressure (without friction) in filter-paper, and kindled by touching it with a hot wire. The flame is dazzling,

* Phosphorus must be cut under water, and very carefully handled, lest it should take fire.

and emits white clouds of anhydrous phosphoric acid. If the piece of phosphorus be too large it will crack the jar.

52. Mix together two volumes of hydrogen and one of oxygen, and with the mixture fill a small jar or tube, which for this experiment should be made of thick glass. On applying a light, the gases combine with a loud explosion, forming water. $H_2+O=H_2O$.

SECTION VIII.

Preparation of Gases which cannot be collected over Water.

53. Although in the preparation of many of the common gases it is most convenient to collect them over water, still there are many cases in which this method is inapplicable, as when the gas is to any considerable extent soluble in water. It is usual in such cases, especially when great purity is necessary, to collect them in tubes or jars over mercury, which is not acted upon by the majority of the gases. For common purposes, however, some of them may be collected by the displacement of common air in dry bottles, and the more the gas differs in density from atmospheric air, the more is this method applicable.

Hydrochloric acid gas and ammonia may be prepared to illustrate the process.

Preparation of Hydrochloric Acid Gas (HCl).*

54. This gas is easily obtained by the action of sulphuric acid on common salt.

To the neck of a dry Florence flask, a bent tube of the form represented in fig. 15, is adapted by means of a perforated cork; and the flask is charged by introducing 300 grains of dry chloride of sodium (NaCl) and adding to it six fluid drachms of strong sulphuric acid. Immediate effervescence takes place, and the bent tube is passed down to the bottom of a *dry* bottle of about a pint capacity, which should be furnished with a greased stopper or glass plate; while the bottle is being filled, the mouth may be loosely closed with a piece of card or paper.†

* The specific gravity of hydrochloric acid gas is 1·247, 100 cubic inches weighing 39·64 grains.

† If common salt be melted in an earthen crucible and poured out upon a

55. Observe the dense fumes which are formed wherever the gas mixes with the air, especially if the atmosphere is damp, owing to the combination of the gas with the aqueous vapour. The bottle may be considered full when the gas has been flowing over from the mouth of the bottle for two or three minutes; the tube should then be slowly withdrawn, and the bottle tightly closed with the stopper or plate. Three or four bottles may be similarly filled with the gas, a gentle heat being applied when the evolution of gas slackens.

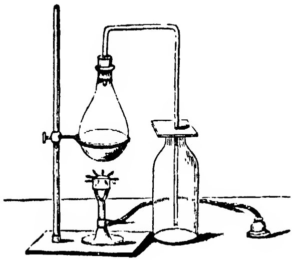


Fig. 15. Preparation of Hydrochloric Acid Gas.

56. The action of strong sulphuric acid upon chloride of sodium, consists in an exchange of one-half of the hydrogen in the former for the sodium in the latter, producing acid sulphate of sodium, which remains in the flask, and hydrochloric acid gas.

$$NaCl + H_2SO_4 = NaHSO_4 + HCl.$$

57. Ascertain the action of the gas on a piece of blue litmus paper.

58. Remove the stopper from one of the bottles, instantly close it again with a dry glass plate (a precaution which is on no account to be omitted, as the stopper might in that case become immovably fixed), and plunge it with the mouth downwards into the water of the pneumatic trough. If the bottle has been well filled, the water will, when the glass plate is removed, quickly rise and nearly fill it, while the unabsorbed residue shows the quantity of common air left in the bottle.*

This experiment must not be made without first removing the stopper, and substituting the glass plate; if it is attempted to take out the stopper while the bottle is

dry stone, it will evolve a very steady supply of hydrochloric acid gas when heated with sulphuric acid. Lumps of sal-ammoniac (hydrochlorate of ammonia) may also be used with advantage.

* Water at common temperatures is capable of dissolving no less than 480 times its own volume of hydrochloric acid. The liquid hydrochloric or muriatic acid of commerce is a solution of the gas in water.

under water, there is great danger of its becoming so firmly fixed, as to be almost incapable of removal, owing to the absorption of the gas by the water, and the formation of a partial vacuum.

59. Test a little of the acid solution obtained in the last experiment, in a test tube, with litmus paper, and afterwards with a few drops of solution of nitrate of silver.* Close the tube with the thumb and shake it violently. Let the precipitate (chloride of silver) settle, pour away the liquid, and treat the precipitate with nitric acid, which will not dissolve it. Pour off the nitric acid, and shake the precipitate with a little ammonia, which will dissolve it at once.

60. Reserve a bottle of the gas for an experiment (66) with ammonia.

SECTION IX.

Preparation of Ammonia Gas (NH_3).†

61. This gas may be collected in a similar manner to the last; but as it is specifically lighter than common air, the bottles in which it is collected must be kept, while filling, with the mouth downwards, the delivering tube passing upwards to the top (fig. 16). The bottles must be perfectly dry. They may be supported upon a perforated wooden block or tin plate, placed upon the ring.

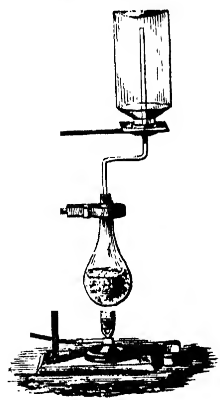


Fig. 16.

62. Mix 300 grains of powdered quick-lime (CaO) with 300 grains of chloride of ammonium (NH_4Cl) in a mortar, transfer the mixture to the flask, and apply a very moderate heat. Chloride of calcium, together with the excess of lime, will be left in the flask, whilst ammonia and water are evolved.‡

To ascertain when the bottle is filled with gas, hold a

* $HCl+AgNO_3=AgCl+HNO_3$.

† The specific gravity of ammonia gas is 0·589, 100 cubic inches weighing 18·288 grains.

‡ $2\ NH_4Cl+CaO=2NH_3+H_2O+CaCl_2$.

piece of moistened turmeric or red litmus paper near its mouth; a strong alkaline reaction will be perceived. When this is the case, slide a greased glass plate under the mouth of the bottle and slowly withdraw it, replacing it immediately by another. The closed bottle should be set aside with its mouth downwards. When three or four bottles have been filled, proceed with the following experiments:—

63. Observe the effect of the gas on a lighted taper slowly thrust up into the inverted bottle: it extinguishes the flame, and at the same time shows a slight tendency to burn with a pale green flame.

64. Place one of the bottles with its mouth downwards under water, remove the glass plate and observe the rapid absorption. That which remains unabsorbed is atmospheric air.*

65. Test the liquid obtained in the last experiment (which is a weak solution of ammonia) with turmeric and reddened litmus paper; the former is turned brown, the latter has the blue colour restored.

66. Invert the reserved bottle of hydrochloric acid (60) over the bottle of ammonia (fig. 17), and cautiously remove the glass plates so as to allow the gases to mix. Dense white fumes, consisting of hydrochlorate of ammonia or chloride of ammonium, are immediately produced, which in a short time collect in flakes, and fall like snow on the sides and bottom of the vessels. In this combination of the hydrochloric acid with the ammonia, considerable heat is evolved. Close the bottles again with glass plates, place them upon the table, and test the gas left in each with blue and red litmus paper, to ascertain if either of the gases was employed in excess. $HCl+NH_3=NH_4Cl$.

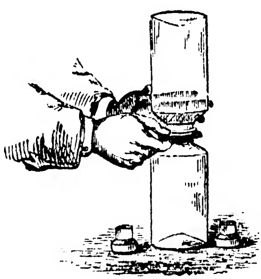


Fig. 17.

* Water at common temperatures is capable of absorbing nearly 700 times its volume of ammonia gas.

CHAPTER III.

DISTILLATION.

SECTION I.

Distillation of Water.

67. Adapt a cork to the neck of a quilled receiver, and bore a hole through it to fit the neck of the retort, which should pass through it for about two inches. When this is done, the apparatus may be fitted up as shown in the figure (fig. 18). The funnel which supplies water for

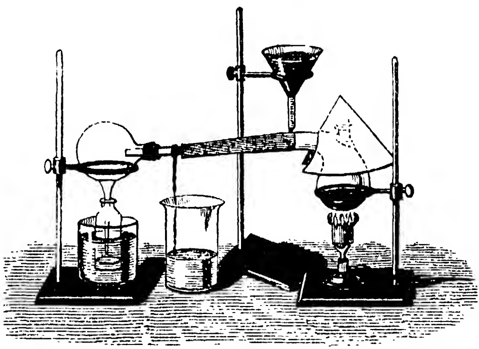


Fig. 18.

cooling the neck of the retort, has its throat partially obstructed by a plug of tow, so that the water may flow only drop by drop; the neck of the retort is covered by a slip of filter paper, cut of such a width as almost completely to encircle the neck, and tenderly laid lengthwise on after being wetted; between the lower end of the paper and the quill receiver a thin fillet of wet tow is bound twice round the glass, and the ends twisted into a tail to carry off the superfluous water, which drops into a basin placed underneath for its reception. The quill of

the receiver passes into a small flask or bottle, which is kept immersed in water during the process, in order to keep it cool.

68. When the apparatus is thus arranged, the retort must be cautiously charged with common water till nearly half full, care being taken that none of it gets into the neck, as it would run down into the receiver and contaminate the distilled water, which would otherwise be pure. The upper part of the body of the retort being then covered with a conical cap of stiff brown paper to prevent loss of heat by currents of air and radiation, the lamp may be applied, care being taken that the ebullition does not go on too violently, lest any of the impure water should splash or boil over into the neck of the retort. The first ounce of water that comes over should be rejected as impure, after which two or three ounces may be distilled for examination.*

69. While the distillation is going on, another portion of the water operated on may be tested, with the view of discovering some of the impurities present in it. Fill four test-tubes† about one-third full of the undistilled water, and add to them respectively a few drops of the following reagents:—

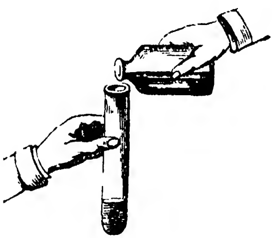


Fig. 19.

(*a*) To the first add a solution of *chloride of barium;* a white precipitate, insoluble in dilute hydrochloric acid,‡ indicates the presence of sulphates, most commonly sulphate of lime.

(*b*) To another portion add a solution of *nitrate of silver.* If any chloride is present (usually chloride of sodium), a white curdy precipitate of chloride of silver will be produced, insoluble in nitric acid, but readily soluble in ammonia. By exposure

* A retort must never be heated before the liquid is introduced, and the flame must be so regulated during the distillation, that the sides of the retort above the level of the liquid may not be too highly heated.

† The tests are of course more delicate when applied to larger quantities of the water, such as a wineglass or tumblerful.

‡ In testing the solubility of a precipitate in any liquid, pour off a small portion into a separate tube for the experiment, reserving the rest for comparison.

to the light this precipitate gradually becomes purple, especially when the water contains organic matter.

(*c*) To the third tube add a little *lime-water;* a white precipitate, soluble in hydrochloric acid, shows that a carbonic acid is present.*

(*d*) To the remaining tube *oxalate of ammonia* may be added, which will give a white precipitate if any lime is present.

70. Test the distilled water in the same way; if pure it will of course furnish no precipitate with any of the re-agents.†

71. Evaporate a few drops both of the distilled and undistilled water on platinum foil or a clean slip of glass: a considerable residue will probably be left by the latter, but no trace of solid matter ought to be observable where the distilled water was evaporated.

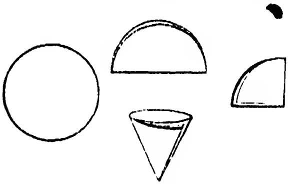


Fig. 20.

72. During ebullition, the water in the retort usually becomes turbid, owing to the formation of a white insoluble powder, which may be separated by filtration when the distillation is over.

To prepare a filter, take a circular piece of white filtering or blotting paper, and fold it neatly, so as to form a cone when opened out (fig. 20). Moisten the paper placed in a funnel with distilled water, and then carefully pour in the liquid to be filtered (fig. 21).

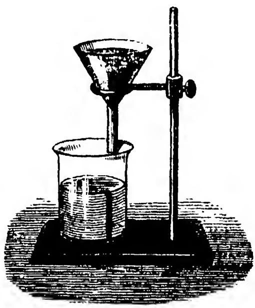


Fig. 21.

When the liquid has passed through, the filter and funnel are placed over another test-glass; the clear solution being reserved for examination (75).

* This test would only detect carbonic acid if present in considerable quantity: its failure must not be assumed to indicate the entire absence of carbonic acid.

† In order to prove the cleanliness of the tube, it is advisable to pour a few drops of the reagent into it, and then to add the water to be tested.

73. A large proportion of the deposit will be found upon the sides of the retort; pour into this two drachms of *dilute hydrochloric acid*, when the deposit will dissolve with effervescence, indicating that it is a carbonate (probably of lime). Rinse the acid round the retort and pour it upon the filter to dissolve the rest of the deposit.

74. Add to a little of the solution thus obtained ammonia in slight excess,* and a little *oxalate of ammonia*: a white precipitate shows the presence of lime. The powder is thus proved to contain carbonate of lime. This carbonate of lime had been held in solution by the excess of carbonic acid contained in the water; when the gas is expelled during ebullition, the carbonate is precipitated.

75. Test the solution filtered from the carbonate of lime in (72) with *chloride of barium*, *nitrate of silver*, *lime-water*, and *oxalate of ammonia*; and compare the results with those obtained in (69), when the water was examined in its natural state. As most of the lime has been separated as carbonate, we may expect to find less of it in solution than before, but more of the sulphates and chlorides, since they still remain dissolved in a more concentrated form.

SECTION II.

Distillation of Hydrochloric Acid.

76. Fit up the apparatus as in the ordinary process of distilling water (67), taking care that the cork is perfectly air-tight; then remove the retort, and introduce through the tubulure 1000 grains† of dry chloride of sodium in coarse powder, taking care that none of the particles fall into the neck of the retort: then adjust the apparatus as before. Measure into the small flask or bottle which is to receive the distilled acid 12 fluid drachms of water, and mark with a file the height at which it stands; and having emptied it, measure into it seven drachms of dis-

* To judge if the ammonia is in excess, close the mouth of the tube with the thumb, and shake it. The excess of ammonia will be perceived by its odour.

† In this and many other of the experiments, small quantities are mentioned to suit the convenience of my class of Practical Chemistry, the lessons being only two hours long. When the products of the experiments are wanted for use, much larger quantities must frequently be employed.

tilled water, which will be increased to 12 drachms by the hydrochloric acid which distils over. During the distillation care must be taken that the quill of the receiver dips just under the surface of this water, which will assist in condensing the acid fumes, some of which might otherwise escape; but the tube need not pass to the bottom of the water, because the solution of hydrochloric acid is the heavier of the two.

77. Into a small evaporating basin pour seven drachms of water, and add gradually to it six drachms of strong sulphuric acid, stirring the mixture with a glass rod. When nearly cool, this dilute acid may be poured carefully into the retort through a small funnel, avoiding any splashing or soiling of the neck. A gentle heat may then be applied, which must be regulated according to the rapidity with which the acid distils over, great care being taken that the mixture does not boil over into the neck of the retort (56).

The distillation may be continued until the liquid in the receiving bottle has increased to twelve drachms of acid, which may be known by the mark previously made.

The acid in the receiver may now be examined as to its purity. Pour a little into a test tube, dilute it with about three times its bulk of water, and add a few drops of a solution of *chloride of barium;* if a white precipitate appears which is insoluble in the acid, it shows the presence of sulphuric acid as an impurity.*

78. Evaporate a few drops of the acid on platinum foil or a clean slip of glass; no trace ought to remain. Any solid residue shows the presence of some saline impurity, caused, probably, by a little of the salt employed having got into the neck of the retort, and been washed down into the receiver.

SECTION III.

Distillation of Ammonia.

79. Prepare the apparatus as in the distillation of water (67).

* Repeat the experiment with the undiluted hydrochloric acid. A crystalline precipitate will be formed which might be supposed to indicate sulphuric acid, but it will be dissolved on shaking with water, for it consists of chloride of barium which is insoluble in strong hydrochloric acid.

Introduce into the retort, through the tubulure, 450 grains of quick-lime in powder, and 530 grains of chloride of ammonium, add five ounces of distilled water, and shake the retort to prevent the lime from caking at the bottom. Measure into the receiving flask or bottle fifteen drachms of water, and mark with a file the height at which it stands; empty it, and pour in two drachms of distilled water for the quill of the receiver to dip into during the distillation.

The distillation may now be commenced, carefully regulating the heat, and continuing it until the distilled liquid reaches up to the file mark in the receiver, when fifteen drachms will have been obtained.

80. Pour a little of the ammonia thus prepared into a test tube, and add to it a few drops of *chloride of barium;* if a precipitate appears, it is owing to the presence either of carbonic or sulphuric acid. To distinguish between them, add dilute *hydrochloric acid* in slight excess; if the precipitate thereupon dissolves, it is due to carbonic acid; if not, to sulphuric.

81. Test another portion of the ammonia with a little *oxalate of ammonia;* if a white precipitate is formed, it is owing to the presence of lime as an impurity.

82. Add to a little of the distilled liquid a slight excess of dilute nitric acid* in a test tube, and add a few drops of a solution of *nitrate of silver;* a white precipitate indicates the presence of a chloride. If a further portion of the ammonia be added, so as to render the liquid alkaline, the precipitate redissolves.

83. If no precipitate occur with any of these tests, evaporate a few drops of the ammonia on a slip of glass or platinum foil, and observe whether any trace of saline impurity is left.

SECTION IV.

Distillation of Nitric Acid.

84. Fit up the apparatus as in the distillation of water (67).† Introduce into the retort 1000 grains of nitrate of

* Enough to destroy the smell of ammonia.

† In preparing this acid upon a larger scale, it would be necessary to avoid the use of cork, which is acted upon by the acid; the neck of the retort would then be merely slipped into that of the receiver.

potash (KNO_3); pour upon it ten drachms of strong sulphuric acid, previously diluted with an equal bulk of water (77), and apply a gentle heat, observing the same precautions as were recommended in the former cases. Nitric acid will distil over, leaving bisulphate of potash (or acid potassium sulphate) in the retort.

$$KNO_3+H_2SO_4=KHSO_4+HNO_3.$$

85. While the distillation is going on, dissolve a few crystals of the nitrate of potash in distilled water, for the purpose of ascertaining its purity.

(*a*) Test a little of the solution with *nitrate of silver;* if any chloride is present, a white curdy precipitate appears, which is insoluble in nitric acid, but readily soluble in ammonia.

If the nitrate employed is contaminated with any chloride, the acid which distils over is sure to contain a little chlorine.*

(*b*) To another portion, add a solution of *chloride of barium;* if any sulphates are present, a white precipitate is produced, which is insoluble in dilute hydrochloric acid.

86. Dissolve 250 grains of nitrate of potash in an ounce of hot water, in an evaporating basin *a*, supported on the *retort stand b* (fig. 22), and observe the gradual formation of crystals as the solution cools. Remove some of these from the liquid, and dry them on filtering paper; then redissolve them in distilled water, and test the solution as before, with *nitrate of silver* and *chloride of barium.* The precipitates, if any, will be less dense than in the previous examination, showing that a partial purification has been effected.

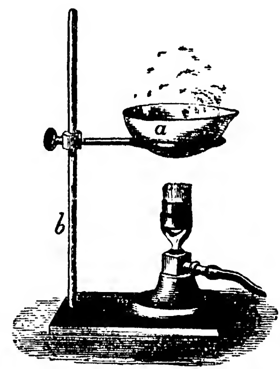


Fig. 22.

87. The distilled nitric acid may now be tested for impurities, but before the test liquids are applied, a portion should be diluted with four or five times its bulk of dis-

* That which distils over at the commencement will be found to contain most chlorine.

tilled water, since the chloride of barium and nitrate of baryta are insoluble in strong nitric acid, and would consequently cause a precipitate, even though no sulphuric acid were present. A portion may then be tested for sulphates and chlorides with *chloride of barium* and *nitrate of silver*.

CHAPTER IV.

GLASS-WORKING.

88. The most convenient form of apparatus for working glass on the small scale, is the water blowpipe, which consists of an upright box, about fifteen inches high, of the form represented in figure 23. It is usually made of zinc or copper, and is divided into two compartments by the plate *a*, which passes down to within about half an inch of the bottom, thus leaving a communication open between the two. The lower end of the tube *b* is closed by a valve opening outwards: the box should be filled about half full of water, and when used, air is blown through the tube *b*. The pressure thus occasioned in the compartment *c*, forces a portion of the water into the next division *d*, where it rises to a higher level than in *c*, and by its superior pressure forces a stream of air through the fine aperture at the extremity of the tube *e*, as long as it continues to stand at a higher level than in *c*. In this way a continuous jet is readily obtained, with much less fatigue to the operator than with the mouth blowpipe.

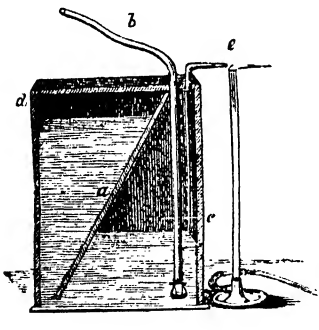


FIG. 23.

89. If the blowpipe flame be examined, it will be found to consist of two distinct parts, which may be called, for

the sake of distinction, the inner *a*, and the outer flame *b* (fig. 24).* The blue point of the inner flame is evidently

Fig. 24. Blowpipe Flame.

surrounded on all sides by the burning gas, the oxygen of the air projected into the flame having been entirely consumed before this point of the flame is reached, so that most substances containing oxygen, placed in it, will be decomposed by the powerful deoxidizing action of the carbon and hydrogen of the combustible gases: on this

Fig. 25.

account the inner flame is usually called the *deoxidizing* or *reducing* flame. The outer flame, on the contrary, is

* Three parts will really be observed; but the innermost dark portion is merely a hollow space filled with the comparatively cool mixture of combustible gas and air.

surrounded on all sides by the external air, so that here there is no excess of combustible or deoxidizing matter, but rather an excess of atmospheric oxygen; hence an oxidized substance may be placed at its extremity without danger of deoxidation, unless such decomposition is effected by the mere heat of the flame, independent of its chemical action; on the other hand, most substances having an attraction for oxygen, placed within its influence, become oxidized, and hence it is usually called the *oxidizing* flame. The hottest part of the blowpipe flame is a very little in advance of the point of the inner blue cone (reducing flame).

90. The English flint glass, of which the tubes and rods commonly in use are made, contains in its composition a quantity of oxide of lead, which, when heated in contact with deoxidizing matter, is very easily decomposed. On this account it is necessary, in heating such glass with the blowpipe, to take care that it does not approach the deoxidizing flame, but is kept at the extremity of the oxidizing (*outer*) flame, otherwise a black stain of metallic lead will be deposited on the surface of the glass. Slight stains of this description may generally be removed by holding the glass for a few seconds in the oxidizing flame; this converts the lead again into oxide, which dissolves in the glass. The German hard glass tubing, which is free from lead, and is much less fusible than the English glass, is far preferable for making tubes, &c., which will be required to withstand a higher temperature.*

Fig. 26.

Fig 27.

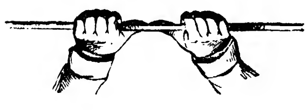


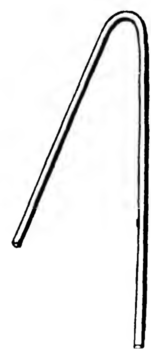
Fig. 28.

* To distinguish tubes containing lead from those which are free from it, hold them so as to look along their *axes*. The ring forming the circumference of the lead-glass is always dark, the other having a greenish tinge.

91. *Make a few glass stirring-rods of lengths varying from five to eight inches* (fig. 26). To do this, a piece of solid rod, long enough to make two stirrers, should be held at a short distance from the extremity of the flame, and gradually brought towards it; a rotating motion being communicated to it by means of the finger and thumb, so that the part where the heat is applied may be uniformly heated all round (fig. 25). When the glass begins to soften it should be gently pulled with both hands, until it assumes the form represented in fig. 27, when it may be removed from the flame; and having been scratched with a file across its narrowest part, may be gently broken asunder (fig. 28).

92. The sharp edges are then held in the flame until they are rounded, so as to form a blunt conical termination to the rod, *a* (fig. 26), whilst the other extremity may be simply fused till it is round and uniform, *b*.

93. *Make a small siphon tube.* Take a piece of narrow tubing* ten or twelve inches long, and hold it diagonally in the upper part of the flaring flame of a gas or spirit lamp (*not in the blowpipe flame*), turning it constantly round, and by gently moving it up and down in the flame, heating two or three inches of the central part of the tube. When the glass begins to soften, apply a gentle pressure with both hands, so as to bend it slowly, and continue to do so until it has assumed the form shown in fig. 29. If the tube be too strongly heated, or the pressure too strongly and suddenly applied, the bend, instead of being round and uniform, will be abrupt and wrinkled, in which case it is very liable to crack, either spontaneously, or when exposed to slight variations of temperature. The extremities of the tube must now be rounded off by being heated to redness for a moment in the flame of the blowpipe. When the glass operated on is at all thick, or of an unequal form, some care is necessary in *annealing* or gradually cooling it: this may be effected by removing it slowly from the flame, and



Fi
Siphc

* About a fourth or a third of an inch in diameter.

then laying it across a piece of tube, so that the hot part does not touch any cold substance, and covering it loosely with paper to prevent too rapid cooling by radiation.

94. *Make a dropping tube* (fig. 30). Take a piece of

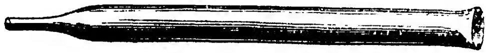


Fig. 30.

narrow tubing, about eight inches long, and moderately thick in the walls. Heat it at about two inches from one end in the lower part of an ordinary flame, so as not to smoke it, rotating it slowly that it may be uniformly heated. When it has softened sufficiently, draw it steadily out to a narrow neck as in fig. 31, taking care to keep it

Fig. 31. Dropping Tube in process of manufacture.

perfectly straight. When cool, scratch the narrow neck with a file, and break it off. Round the sharp edges of the fracture by exposing them for an instant to the extreme point of the blowpipe flame, and fuse the edges of the wider orifice at the other end, so that it may be closed air-tight with the finger.

95. *Seal a small tube at one end* (fig. 32). Take about six inches of narrow tubing, soften it in the outer blowpipe flame, at about two inches from one end, and draw it out quickly to a long thread (fig. 33). Direct the extreme point of a

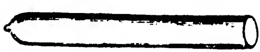


Fig. 32.

Fig. 33. Sealed Tube in course of manufacture.

sharp flame to *a*, and draw it off so as to leave the end neatly rounded. Anneal it by withdrawing it slowly from the flame.

96. *Make a few test-tubes.* A piece of tube may be taken about half or five-eighths of an inch in diameter, and eight or ten inches long, which will serve for two test-tubes.

In cutting off a piece of wide thin tubing, such as is employed for test-tubes, it is advisable to carry the file-

mark round the tube, which is then to be held in a cloth and broken by a combined pulling and breaking movement of the hands. If the end of the broken tube exhibit a crack, this must be led round the tube by applying a red-hot iron point (the end of a file heated by the blowpipe flame will do) a little in advance of it. A glazier's diamond is very useful for cutting thin tubes.

The central portion must be heated in the manner described for heating glass rod (91), and gradually drawn out, the tube being constantly turned round, when it will assume the form shown in fig. 34. The heat should now

Fig. 34.

Fig. 35.

Making Test-Tubes.

be applied to the part of the tube marked *a*, and the other piece gradually drawn out, care being taken not to fuse the thin thread of glass, which is formed, and which connects the two parts of the tube (fig. 35), until the base of the tube has become round and uniform; when this is the case, and the connecting thread has become very thin, the heat may be applied to the point where it joins the tube, when it will instantly fuse and separate, leaving the tube in an almost finished state. There will generally be found at the bottom of the tube, however, a small lump, more or less distinct, formed by a portion of the thread having fused into it; to remove this, again heat the round end red-hot for a short time, until the lump disappears. On removing the tube from the flame, blow air gently into it, for the purpose of swelling out the bottom to its previous round form (fig. 36), as it usually collapses and flattens while in a state of fusion.

Fig. 36.

The other portion of the tube may now be finished in a similar way, by applying the heat to the point *b*, and drawing off the irregular termination until the thread of glass is sufficiently attenuated to be removed.

When it is required to make a test-tube of a piece of tubing only long enough for one, all that is necessary is to

melt on to one end another piece of waste tubing or rod, to serve as a handle, after which the end may be drawn off, as in the former case.

Fig. 37.

To complete the tube, the open end must be spread out a little, as shown in fig. 37, so as to form a kind of border. This is done by softening the end in the blowpipe flame, and then, by means of a thick iron wire, or the smooth end of a file, introduced and carried round the opening, the edge is uniformly pressed outwards.

97. *Blow some small bulbs.* Take a piece of narrow glass tube, sealed at the end as described in (95); heat the end for about half an inch as strongly and uniformly as possible; and then, having removed it from the flame, hold it horizontally and blow air into it until the pressure forces the softened glass to expand, which it will do in the form of a round bulb (fig. 38) if the heat has been properly applied, and the tube be kept constantly turned round while in the hand. This latter precaution is absolutely necessary, as the softened glass would otherwise bend with its own weight in one direction, thus destroying the proper form of the tube.*

Fig. 38.

98. *Make a funnel tube.* Blow a bulb of considerable size on the extremity of a piece of strong tube about a quarter of an inch in diameter. Heat a spot upon the bulb immediately opposite to the point at which the tube enters it, and blow suddenly into it until it bursts; break away the superfluous thin glass with the fingers, and finish off the border with the blowpipe flame and the heated end of a file.

Fig. 39. Funnel Tube.

99. *Cement together two tubes of equal diameter.* The ends to be joined must be cut perfectly level; the edges are brought exactly together, the tubes being kept in the same straight line, and the glass is gradually softened by rotating the tube slowly in the outer flame; a gentle pressure may be applied to perfect the joint, and a little care in

* In order to blow a large bulb upon a thin tube, the glass must be allowed to thicken at the sealed end by keeping it for some time in the flame.

moulding it will soon render it perfectly uniform; it should be well annealed by withdrawing it slowly from the flame. When the glass is thin, the edges which are to be united may be spread out a little, as shown in fig. 40, by means of a wire or file, when the joint will be stronger than it would otherwise be.

Fig. 40. Tubes united.

100. *Cement together two tubes of unequal diameter.* When it is required to join a narrow tube to a wider one, it is necessary to draw out the latter in the blowpipe flame until a portion of it is contracted to the diameter of the former; then with a file it is divided at that point of equal diameter, and cemented to the smaller tube in the same way as in the previous case. Sometimes, when the glass is thin, it is advisable to widen the extremity of the smaller tube, so as to overlap the other (fig. 41), which is readily done by means of an iron wire.

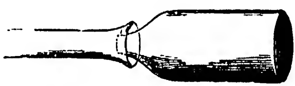


Fig. 41. Tubes united.

In this operation, it is always advisable to maintain the junction in the flame for some little time, to allow of the complete amalgamation of the two portions of glass; and as the tendency to collapse is greater the longer it is fused, it will generally be found necessary to blow it out slightly.

101. *Prepare tubes for a washing bottle.* The tubes required for this purpose are of the form shown in fig. 42, the upper end of the longer one being drawn out so as to leave only a small aperture (94).

When the bottle is prepared and filled with water, a small stream of water may be forced through this tube by blowing air down the shorter one: it is of great service in washing precipitates on a filter.

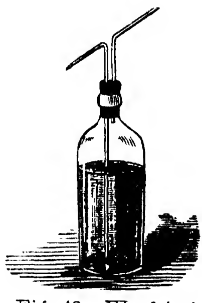


Fig. 42. Washing Bottle.

102. *Prepare tubes for a sulphuretted hydrogen (hydrosulphuric acid) apparatus.* The form of apparatus used for generating hydrosulphuric acid, and passing it into

water or saline solutions, is represented in fig. 43. The tubes, *d* and *f*, are bent in the naked flame (93), the extremities being afterwards slightly fused with the blowpipe in order to round off the sharp edges. The straight tube *g*, is connected with the bent tube *f*, by a tube of vulcanized india-rubber.

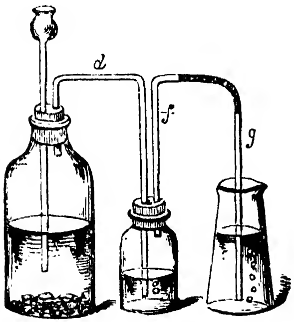


Fig. 43. Sulphuretted hydrogen Apparatus.

103. *Seal a few tubes hermetically at both ends.* This is an operation of very frequent use in the laboratory, as it furnishes the most convenient and efficient means of preserving small specimens of many rare substances, especially such as are volatile; and is often required in effecting chemical changes at elevated temperatures and pressures.

Fig. 44.

The tube is first sealed at one end, precisely as if it were intended for a test-tube (96): the liquid or other substances for which it is designed is then introduced, as soon as the tube is quite cold, care being taken that the upper part of the tube is not wetted or soiled. The flame of the blowpipe is now directed to the portion of the tube a little above that intended for the sealed end, and when sufficiently soft it is drawn out to a capillary tube, and allowed to cool: it may afterwards be sealed by fusing the lower part of the capillary tube *a* (fig. 44), by momentary contact with the flame.*

In this way seal a little sulphur in a tube without melting or volatilizing any of it, the sulphur being within an inch and a half of the upper end.

104. *Seal some water hermetically in a tube.* Having sealed the tube at one end, and drawn it partly out at the other, leaving a rather wide neck, while it is cooling make

* When the tube is required to withstand high pressures, the sealing of both ends must be effected very slowly, so that the glass may remain as thick there as at any other part of the tube.

a dropping tube as in (94), and introduce a little water into the sealed tube without wetting its sides.

Then draw out the capillary neck, and when cold, seal it as before, leaving not more than the space of an inch between the upper end and the surface of the water.*

CHAPTER V.

EXPERIMENTS WITH THE MOUTH BLOWPIPE.

105. Before proceeding to any blowpipe experiments, it is necessary to acquire the power of keeping up a constant and unintermitting blast of air from the mouth, as without this it is impossible to raise the heat to a sufficient degree of intensity. The habit is readily acquired, and when once attained, the mouth and lungs will be found to do their work almost mechanically, without any sustained effort on the part of the operator.

The learner may first observe that on closing the lips he can still without any difficulty breathe through the nostrils: let him now distend the cheeks with air, and he will find that he can breathe through the nostrils for a length of time, still keeping the cheeks distended. He may next introduce the mouth-piece of the blowpipe between his lips, and having puffed out his cheeks with air, let him breathe freely through the nostrils, at the same time allowing the distended cheeks to force a current of air through the blowpipe. The cheeks thus play the part of an elastic bag, which, if connected with the blowpipe, and distended with air, would force air through it as long as the tension of its stretched sides exerted sufficient pressure.

106. Seal a few tubes (fig. 32) at one end, for use in some of the following experiments. The tubing employed for this purpose should be about a quarter of an inch in diameter, and it may be scratched with a file, and broken into pieces about four inches long, each of which will serve for two tubes. The glass of which it is made should not contain lead. The sealing should be effected in the

* When volatile liquids are sealed in tubes, the whole of that portion of the tube which contains the liquid may be surrounded with one or two folds of wet filter paper.

manner already described (95). The position of the operator is shown in fig. 45. It is not necessary to be particular about rounding off the ends of these tubes, but they should be carefully annealed by withdrawing them gradually from the flame. In using one of these tubes for an experiment, it should first be heated in the common flame;† the blowpipe flame being afterwards applied, if the former does not give enough heat: the tube frequently cracks if the blowpipe flame be suddenly applied.

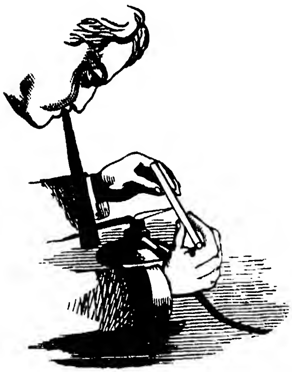


Fig. 45.

These tubes may be most conveniently held by a narrow strap of folded paper passed round the upper part.

The following tests are arranged in alphabetical order, for convenient reference. The student is recommended to try each experiment upon a *known* substance before commencing the examination of unknown substances.‡

Fig. 46. Heating with Blowpipe on Charcoal.

*107. **Aluminium.** (Test some *alum.*) A compound containing when placed in a shallow cavity scooped in a piece of charcoal, and heated in the blowpipe flame (fig. 46), will give a white infusible mass of alumina. On moistening this with a drop or two of solution of nitrate of cobalt, and again strongly heating

† If a gas flame be employed, the tube must be held in the lower blue portion, so as not to be smoked.

‡ The asterisk (*) prefixed to a test designates it as a good example to be selected by the student who intends only to acquaint himself with the general method of testing with the blowpipe.

it, a fine blue mass is obtained, which is a compound of alumina with oxide of cobalt.†

A fragment of the bowl of a tobacco-pipe may be used instead of charcoal in this experiment.

*108. **Ammonia.** (Test some *chloride of ammonium.*) Many compounds of ammonium or ammonia, when heated, in small quantity, in a tube sealed at one end (106), will be entirely converted into vapour and deposited again as a white coating upon the cooler portion of the tube. (The compound is then said to *sublime* unchanged.)

109. (Test some *chloride of ammonium.*) All compounds of ammonium or ammonia, when mixed with about an equal bulk of carbonate of soda, and heated in a tube sealed at one end (106), will yield vapour of carbonate of ammonia which can be recognised by its odour. The mouth of the tube should be slightly inclined downwards (fig. 47) to permit the escape of water, which would otherwise run back and split the tube.

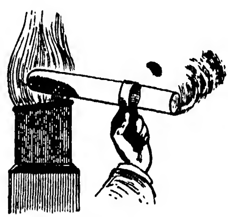


Fig. 47. Small Tube heated in flame.

110. **Antimony.** (Test some *sulphide of antimony.*) Compounds containing antimony, when placed in a shallow cavity on charcoal, covered with a little carbonate of soda, and heated before the blowpipe so that the point of the (inner) reducing flame may touch the specimen, whilst the (outer) oxidizing flame sweeps over the charcoal beyond (fig. 46), will yield globules of metallic antimony which will gradually pass off in vapour if retained too long in the flame. The surface of the charcoal around the oxidizing flame becomes coated with a white incrustation of oxide of antimony. If one of the globules be removed with the point of a knife, placed upon a hard surface, and pressed with the knife-blade, it will be found to possess the brittleness characteristic of metallic antimony.

† This is not a perfectly conclusive test for alumina, since some phosphates and other substances exhibit a similar behaviour.

In blowpipe tests with nitrate of cobalt, care must be taken to employ a very high temperature. A moderate heat produces the blue anhydrous nitrate of cobalt, which a higher temperature converts into black oxide of cobalt, and this requires an intense heat to make it combine with alumina, &c.

1. **Arsenic.** (Test some *arsenious acid.*) On heating a minute quantity of arsenious acid in a small tube sealed at one end, it will be entirely converted into vapour, and if the upper part of the tube be warmed by drawing it once or twice through the flame before heating the arsenious acid, the latter will condense slowly upon the glass, forming a *sublimate* of minute shining crystals, which appear as octahedra (fig. 48) when magnified.

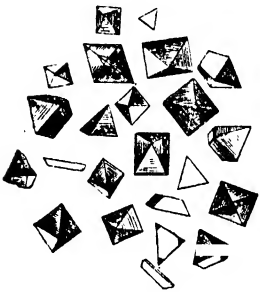


Fig. 48. Arsenious Acid.

*112. (Test some *arsenious acid.*) Any compound containing arsenic, when heated on charcoal before the blowpipe (fig. 46), will evolve an odour of garlic.

*113. (Test some *arsenious acid.*) Any compound containing arsenic will yield a sublimate of metallic arsenic when heated in a tube with carbonate of soda and charcoal.

Dry a little powdered carbonate of soda (upon a strip of tin plate or a piece of thin glass) at a moderate heat, and mix it with about one-third of charcoal powder (scraped from a piece of charcoal). Add to the mixture a very minute quantity of the compound to be tested, introduce it into a small tube sealed at one end, cleanse the upper part of the tube with a roll of paper (or a lucifer-match stick), shake the mixture a little to one side of the tube (fig. 49) to allow the passage of the vapour, and apply heat, first with the lower part of the ordinary flame, and finally with the blowpipe, holding the mouth of the tube a little downwards (fig. 47). A shining black or grey crust of metallic arsenic will be formed upon the upper part of the tube (fig. 49) at *a*. An odour of garlic will probably be perceived at the orifice.

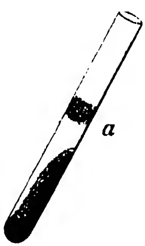


Fig. 49. Crust of Arsenic.

Cut off, with a file, the portion of the tube upon which the deposit has formed, wrap it in a piece of paper, break

it into small fragments, and heat these gently in another small tube, the upper part of which has been first warmed. The oxygen of the air in the tube will convert the arsenic into arsenious acid, condensing in brilliant octahedra on the upper part.

*114. **Barium.** (Test some *barium chloride.*) Any compound containing barium or baryta, well moistened with hydrochloric acid, and intensely heated on platinum wire at the point of the (inner) reducing flame, will impart a bright green colour to the outer flame.†

115. For this purpose, take a piece of thin platinum wire which has been fused into a glass handle (fig. 50).

Fig. 50. Straight wire in glass handle.

Moisten the extremity of the wire with hydrochloric acid, and heat it at the point of the (inner) reducing flame till it no longer tinges the outer flame. Again moisten it with hydrochloric acid, take upon it a minute quantity of the substance to be tested, and expose it to the point of the inner flame.‡

The flame of a Bunsen burner (fig. 85) may be employed instead of a blowpipe flame.

The presence of sodium interferes with this test, on account of its intense yellow flame.

116. **Bismuth.** (Test some *oxide of bismuth.*) Compounds containing bismuth, when placed in a shallow cavity on charcoal, covered with carbonate of soda and exposed to the (inner) reducing blowpipe flame (fig. 46), furnish globules of metallic bismuth, whilst that part of the charcoal which has been traversed by the oxidizing flame becomes covered with a yellow incrustation of oxide of bismuth. If one of the globules, after cooling, be placed upon a hard surface and pressed with a knife-blade, it will first flatten and then break up into small fragments.

† Copper also gives a green flame, but of a different tint: so does boracic acid. The greenish tint imparted by zinc is sometimes mistaken for barium.

‡ Hydrochloric acid is used because the chlorides of the metals are easily vaporized at high temperatures. The vapour is reduced to the metallic state by the heated hydrogen of the flame, and then burns in the outer flame with its characteristic colour.

A compound of bismuth heated with a mixture of equal parts of potassium iodide and sulphur, on charcoal, yields a red incrustation of bismuth iodide. (Von Kobell.) Cuprous iodide answers better than potassium iodide. (Hutchings.)

For this and many other tests depending upon the production of an incrustation, a piece of aluminium plate, as originally recommended by Ross, may be substituted for charcoal with great advantage.

117. **Boracic acid.** (Test some *boracic acid.*) When boracic acid, or a borate, is mixed with some fluor spar and bisulphate of potash, and heated upon a loop of platinum wire (fig. 51) at the point of the inner flame,

Fig. 51.

fluoride of boron will be formed and will impart a green colour to the outer flame.

Borax does not respond well to this test on account of the yellow flame produced by the sodium; but it may be recognised by its *intumescence*, or swelling up, when heated on a loop of platinum wire, and by its fusing to a glass which remains perfectly transparent on cooling.†

118. **Calcium.** (Test some *marble*, carbonate of lime.) Any compound containing calcium or lime, when moistened with hydrochloric acid and heated upon a piece of platinum wire (fig. 50) at the point of the inner flame, will impart a red colour to the outer flame. After the specimen has ceased to colour the flame, it will regain the property when moistened with hydrochloric acid. The presence of sodium interferes with this test (115).

119. **Carbon.** (Test a small fragment of *wood.*) Many compounds containing carbon (*organic matters*), when heated in a small tube sealed at one end (fig. 47), become charred or blackened from the separation of carbon, and at the same time evolve vapours which are frequently inflammable and possess peculiar odours from which the nature of the substance can be inferred.

Carbon in the free state (charcoal, for example,) may be identified by its burning away with a steady glow when heated on a piece of iron or platinum plate, the blowpipe flame being directed upon the under surface of the plate.

† If a platinum wire be moistened with glycerine, and a powdered borate taken upon it, a distinct green tinge will be imparted to the flame, when the glycerine is kindled. Even borax exhibits this reaction.

120. **Carbonic acid.** (Test some *carbonate of soda.*) Carbonates, mixed with bisulphate of potash, and gently heated in a small tube sealed at one end, give rise to a violent effervescence caused by the escape of carbonic acid gas.

A carbonate may also be known by its effervescing when moistened with hydrochloric acid.

121. **Chloric acid.** (Test some *chlorate of potash.*) A chlorate, when heated on charcoal (fig. 46) causes *deflagration*, due to the rapid combustion of the charcoal in the oxygen furnished by the chlorate.

122. A chlorate, heated in a small tube sealed at one end (fig. 47), will evolve oxygen, recognised by its power of kindling into flame a spark upon the end of a match.

123. When a chlorate is mixed with bisulphate of potash, and heated in a tube sealed at one end (fig. 47), it evolves chlorine, which may be known by its odour and its bleaching test-papers.

124. **Chlorine.** (Test some *chloride of sodium.*) Chlorides, mixed with bisulphate of potash, and heated in a small tube sealed at one end (fig. 47), give off pungent vapours of hydrochloric acid, which fume strongly in damp air, and redden blue litmus paper. If a little nitre be added, chlorine is evolved which bleaches the test-paper.

*125. **Chromium.** (Test some *chromate of potash.*) Compounds containing chromium, when fused with borax-glass in the (outer) oxidizing flame, give a yellowish-green transparent glass, which becomes of a pure emerald green colour in the (inner) reducing flame.

126. To apply this test, take a thin platinum wire sealed into a glass handle, and bend the end into a small loop, not larger than the transverse section of the blowpipe flame (fig. 51). Heat the loop to redness, and dip it into powdered borax; heat the borax in the blowpipe flame till it fuses into a perfectly colourless glass; whilst this is red-hot, touch a very minute particle of the compound to be tested with it, and heat the glass, with the adhering particle, in the oxidizing flame. Having observed its colour when cold, repeat the experiment in the reducing flame (*see* 284).

127. **Cobalt.** (Test some *nitrate of cobalt.*) A compound containing cobalt, when fused with a bead of borax (126) on platinum wire, gives a beautiful blue glass in both

flames. It is sufficient to moisten the bead with the solution containing cobalt, and if this be weak, the moistening may be repeated.

128. **Copper.** (Test some *sulphate of copper.*) Copper compounds, when fused with carbonate of soda in a cavity on charcoal, in the (inner) reducing blowpipe flame (fig. 46), yield a red mass of metallic copper which may be fused (if not too large) into a globule, in the focus (or extreme point) of the inner flame. When the globule is pressed with a knife-blade upon a hard surface, the malleability and red colour of the copper will be recognised.† There is no incrustation of oxide of copper upon the charcoal, but a film of grey ash, such as is always left when charcoal is burnt.

129. When a minute quantity of a compound of copper is taken up on a hot bead of borax-glass (126) and fused in the outer flame, it yields a glass which is green while hot, and becomes blue on cooling. When the bead is heated in a very powerful reducing flame, it becomes colourless, the copper being reduced to the metallic state, and fused on to the wire; but in a feebler flame, the bead acquires an opaque brown-red colour, from the presence of suboxide of copper and diffused metallic copper.

130. If a compound of copper moistened with hydrochloric acid be exposed, on the point of a platinum wire (fig. 50) to the reducing flame, it will impart a bluish-green colour to the oxidizing flame.

131. **Fluorine.** (Test some *fluor spar*, fluoride of calcium.) When a fluoride is mixed with bisulphate of potash and heated in a tube sealed at one end (fig. 47), it evolves very pungent vapours of hydrofluoric acid, which fume strongly in damp air, and corrode the glass, giving a dim and greasy appearance to the sides of the tube.‡

132. **Iodine.** (Test some *iodide of lead.*) An iodide, when mixed with bisulphate of potash, and heated in a small tube sealed at one end (fig. 47), evolves violet

† When sulphide of copper is tested in this manner, it is advisable to *roast* it in the oxidizing flame as long as sulphurous acid is evolved, before adding the carbonate of soda. If this be omitted, the reduced globule is likely to be brittle, from the presence of sulphur.

‡ Fluoride of calcium (fluor-spar) may be identified by mixing it with an equal volume of sulphate of lime, and heating on a loop of platinum wire, when it fuses easily to a clear glass which becomes opaque on cooling.

vapours of iodine, having a peculiar odour, and condensing in black scales on the cooler part of the tube.

133. **Iron.** (Test some *sulphate of iron.*) When a compound of iron is fused, in minute quantity, with a bead of borax (126), it colours the glass brownish-yellow in the outer flame, the colour fading rapidly as the bead cools, and becoming bottle-green in the inner flame.

*134. **Lead.** (Test some *red oxide of lead.*) Compounds of lead, fused with carbonate of soda, in a shallow cavity scooped near to one end of a flat surface of charcoal (fig. 46), yield, in the reducing flame, globules of metallic lead, whilst the surface of the charcoal around the oxidizing flame becomes covered with a yellow incrustation of oxide of lead. If the globules be removed with the point of a knife, and pressed with a knife-blade upon a hard surface, they will be known to be lead by the ease with which they may be flattened out and cut, and by their making a dark streak upon paper.

135. **Lime.** *See* Calcium (118).

136. **Magnesium.** (Test some *sulphate of magnesia.*) When compounds of magnesium are heated on charcoal at the point of the inner flame, they generally give a white infusible mass, which assumes a delicate pink colour when moistened with nitrate of cobalt and again intensely heated, a compound of magnesia with oxide of cobalt being formed (*see* note to 107).

*137. **Manganese.** (Test some *binoxide of manganese.*) If a very minute particle of a compound of manganese be taken up on a hot bead of borax (126), and fused in the (outer) oxidizing flame, it gives an amethyst purple or violet colour to the glass, which fades and finally disappears altogether in the inner flame.

138. When fused with carbonate of soda, on platinum wire, in the outer flame, compounds of manganese give an opaque mass (manganate of soda) which is green while hot and becomes blue on cooling. In the inner flame, the colour disappears.

139. To make a bead of carbonate of soda, the loop of wire (fig. 51) should be moistened in the mouth and dipped into the powdered carbonate; if the fused mass does not fill the loop, let it cool, moisten it and dip again.

*140. **Mercury.** (Test some *calomel.*) When a very minute quantity of a compound containing mercury is

mixed with dry carbonate of soda (113), and heated in a small tube sealed at one end (fig. 47), the open end being slightly inclined downwards, a grey deposit of minute globules of mercury will be formed upon the cool part of the tube. By rubbing the crust with a slender glass rod† or a match, the globules will be made more evident.

The upper part of the tube should be thoroughly cleansed with a roll of paper or a match after introducing the mixture.

*141. *Corrosive sublimate* (mercuric chloride) is easily distinguished from *calomel* (mercurous chloride) by the action of a gentle heat upon them in small tubes sealed at one end (fig. 47). Corrosive sublimate fuses before subliming, and crystallizes in needles on cooling, which are often seen projecting from the sides on looking down the tube. Calomel sublimes without fusing, and seldom appears crystalline.

142. **Nickel.** (Test some *sulphate of nickel.*) Compounds of nickel, fused, in minute quantity, with a bead of borax (126) in the outer flame, furnish a brownish-yellow glass, which becomes dusky and grey in the inner flame. If the hot bead be made to take up a very small particle of nitrate of potash, and again exposed to the outer flame, it acquires a purple colour.

*143. **Nitric acid.** (Test some *nitrate of potash.*) If a nitrate be placed in a cavity on charcoal, and heated with the blowpipe flame, *deflagration* will be caused by the combustion of the charcoal in the oxygen furnished by the nitrate.

144. Nitrates, mixed with about an equal quantity of bisulphate of potash, and heated in a small tube sealed at one end (fig. 47), give off red-brown vapours of nitric peroxide which have a characteristic odour.

145. **Nitrogen.** (Test some *isinglass* or *gelatine.*) Organic substances containing nitrogen, when heated in a small tube sealed at one end (fig. 47) evolve offensive vapours which are usually alkaline to test-papers, from the presence of ammonia.

146. **Organic matter.** (*See* 119, 145.)

147. **Oxygen.** (*See* 122.)

148. **Phosphoric acid.** (Test some *phosphate of*

† Easily made by drawing out a piece of glass rod in the blowpipe flame.

lime.† Mix the compound to be tested for phosphoric acid with two or three parts of dried boracic acid, and fuse it in a cavity scooped on charcoal (fig. 46). Whilst the mass is still hot, thrust into it about half an inch of very thin iron wire (of which twelve inches weigh one grain), and heat it for two or three minutes in a powerful reducing flame. If phosphoric acid be present, a hard silvery brittle metallic globule of iron containing phosphorus will be formed, which may be extracted from the fused mass by wrapping it in stout paper and striking it with a hammer upon an anvil.

148*a*. If phosphate of lime be moistened with strong sulphuric acid, and heated on platinum wire at the point of the inner blowpipe flame, a part of the phosphoric acid is reduced, and the vapour of phosphorus burns with a peculiar livid greenish colour in the outer flame.

149. **Potassium.** (Test some *nitrate of potash.*) Compounds containing potassium, when moistened with hydrochloric acid, and heated on the point of a platinum wire (fig. 50) in the reducing flame, impart a transient violet colour to the oxidizing flame. If sodium be present, the yellow colour of its flame will obscure the violet tinge.‡.

150. **Silicic acid.** (Test some white sand.) Make a bead of carbonate of soda in a loop of platinum wire (139), and observe that it becomes opaque on cooling. Heat it again to redness, dip it into the compound to be tested, and heat it again. Silicic acid will dissolve with effervescence, from expulsion of carbonic acid. By repeated fusion with successive additions of silicic acid the bead will be made transparent on cooling.§

151. Make a bead of microcosmic salt in the loop of platinum wire. The salt is a phosphate of soda, ammonia, and water; when heated, it loses the two last, and is converted into a transparent glass of metaphosphate of soda.‖

† This experiment requires a very steady and powerful application of the blowpipe flame, and forms a good criterion of the skill of the operator. Borax may be substituted for boracic acid, but is far less convenient because of its intumescence.

‡ By observing the flame through a piece of cobalt-blue glass, the yellow sodium flame may be cut off, and the potassium flame then becomes visible.

§ If a sulphate (or sulphur in any form) be present, the bead will have a brown colour, especially when hot, in the reducing flame, from the formation of sulphide of sodium.

‖ $NaNH_4HPO_4 = NaPO_3 + NH_3 + H_2O$.

Take a little silica upon the hot bead, and observe that it floats about undissolved in the fused glass, and may be seen as an opaque mass when the bead cools.

152. **Silver.** (Test some *chloride of silver.*) Any compound containing silver, fused in a cavity on charcoal with carbonate of soda (fig. 46), will yield a white mass of metallic silver, which fuses into a brilliant globule in the focus of the flame, and does not tarnish in the outer flame. When pressed with a knife upon a hard surface, the globule will flatten out, but requires considerable pressure. There will be no incrustation of oxide of silver upon the charcoal, but the usual film of ash will be seen, and perhaps a little white deposit of chloride of sodium.

153. *Extraction of silver from lead by cupellation.* Scoop out a large shallow cavity in a piece of charcoal *a* (fig. 52) and fill it with powdered bone-ash (phosphate of lime) rammed very tightly down (*b*); place a small fragment of argentiferous lead upon it, and expose it for two or three minutes to the extreme point of a steady oxidizing flame, taking care to hold the charcoal quite horizontal, or the globule of lead will run off. Observe that the oxide of lead, as it is formed, fuses and is absorbed by the porous phosphate of lime, leaving a very small bead of silver which no longer tarnishes or diminishes in the outer flame, even though placed upon a fresh bed of bone-ash.

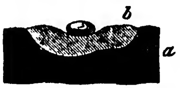


Fig. 52.

*154. **Sodium.** (Test some *carbonate of soda.*) Compounds containing sodium, even in very minute proportion, when heated on platinum wire in the inner blowpipe flame, impart a fine golden yellow colour to the outer flame. A clean platinum wire drawn through the fingers acquires enough chloride of sodium from the skin to tinge the flame yellow.†

*155. **Strontium.** (Test some *nitrate of strontia.*) Any compound containing strontium, when moistened with hydrochloric acid, and exposed on the point of a platinum wire (fig. 50) to the inner flame, imparts a carmine red colour to the outer flame. Careful comparison is

† Most substances contain enough sodium to impart a yellow tinge to the outer blowpipe flame, so that the existence of sodium as an essential constituent of a substance under examination, can be inferred only when the golden flame is very decided and lasting, and when there is some corroborative evidence from other sources.

required to prevent calcium from being mistaken for strontium by this test (118).

156. **Sulphur.** (Test some flowers of *sulphur*.) Free sulphur may be recognised by heating it gently in a small tube sealed at one end (fig. 47), when it easily fuses to a yellow liquid, becoming brown as the heat is increased, and yielding a brown vapour, condensing in brown and yellow drops upon the cool part of the tube. If moisture be present in the sulphur, a smell of sulphuretted hydrogen will be perceived at the mouth of the tube.

157. When sulphur is heated on a platinum or iron plate, it burns with a blue flame, evolving the odour of sulphurous acid.

The sulphur in many metallic sulphides may be recognised by this test, especially if it be applied in a small tube, about four inches long, open at both ends, and slightly bent in the middle to prevent the substance from slipping out.

158. (Test some *sulphate of baryta*.) Any compound containing sulphur, mixed with carbonate of soda and charcoal powder, and exposed to the reducing flame in a cavity scooped on charcoal (fig. 46) will yield a mass containing sulphide of sodium. If this be placed upon a clean silver coin, and moistened with a drop of hydrochloric acid, it will evolve the odour of sulphuretted hydrogen, and leave a black stain of sulphide of silver upon the coin.

In this experiment the sulphate of baryta is decomposed by the carbonate of soda, yielding carbonate of baryta and sulphate of soda. This is deoxidized by the charcoal, aided by the reducing flame, and becomes sulphide of sodium, which the hydrochloric acid converts into chloride of sodium and sulphuretted hydrogen.†

159. **Sulphuric acid.** (*See* 158.)

160. (Test some *sulphate of iron*.) Many sulphates, heated in a small tube sealed at one end (fig. 47) evolve a mixture of sulphurous and sulphuric acid, the former being recognised by its odour, and the latter by its dense white fumes. Blue litmus paper is very strongly reddened by the vapours.

161. **Tin.** (Test some *binoxide of tin*.) Compounds containing tin, fused in a cavity on the charcoal (fig. 46) in the reducing flame, with successive additions of car-

† When a coal-gas flame is used for this experiment, sulphur is often derived from the gas. A spirit-lamp flame avoids error from this source.

bonate of soda, will eventually yield globules of metallic tin, the brilliancy of which is obscured on withdrawing them from the flame, by the formation of a film of oxide. When a globule of tin is pressed under a knife-blade on a hard surface, it flattens out more easily than silver. There is no marked incrustation on the charcoal in this experiment.

162. Coarsely powdered cyanide of potassium may, with great advantage, be substituted for carbonate of soda in the preceding experiment.

163. **Zinc.** (Test some *sulphate of zinc.*) Compounds containing zinc, heated on charcoal before the blowpipe, generally yield an infusible mass which is yellow while hot, and becomes white on cooling. By moistening the mass with nitrate of cobalt and again intensely heating, a bright green compound of the oxides of zinc and cobalt is produced. (*See* note to 107.)

164. When a compound of zinc is fused on charcoal with carbonate of soda in the reducing flame (fig. 46) the metal is vaporized as fast as it is reduced, and gives an incrustation of oxide, which is yellow while hot and white on cooling.

The blowpipe tests for zinc are much more distinct when a fragment of the bowl of a tobacco-pipe is used as a support instead of the charcoal.

SYSTEMATIC EXAMINATION OF UNKNOWN SUBSTANCES WITH THE BLOWPIPE.†

165. Heat a little of the substance in a cavity on charcoal (fig. 46).

Deflagration indicates a *nitrate* or *chlorate* (122, 123, 144).

Odour of sulphurous acid indicates *sulphur* (a *sulphide*, or a *sulphate* (156, 157, 158, 160).

Odour of garlic indicates *arsenic* (111, 113).

White incandescent‡ mass. Moisten with nitrate of cobalt, and heat again, to detect *zinc* (green), *aluminium* (blue), or *magnesium* (pink). (*See* 107, note.)

† A list of substances which may be examined as exercises in this systematic course will be found in the Appendix.

‡ Incandescent, evolving a brilliant white light when heated in the blowpipe flame.

166. Fuse the substance with carbonate of soda on charcoal† (fig. 46).

	Metallic bead.	Incrustation.
Antimony.	**Very brittle.**	**White.**
Bismuth.	**Brittle.**	**Yellow.**
Copper.	**Red, malleable.**	
Lead.	**Malleable.**	**Yellow.**
Silver.	**Malleable.**	
Tin.	**Malleable.**	
Zinc.		**Yellow** while hot. **White** when cold.

167. Test for *sulphur* in the fused mass from the preceding experiment, as in (158).

168. Take the substance, in very minute successive portions, upon a red-hot bead of borax-glass, and fuse in the outer and inner blowpipe flames (126).

	Outer flame.	Inner flame.
Chromium.	**Yellow-greenish glass.**	**Emerald-green glass.**
Cobalt.	**Blue** „	**Blue** „
Copper.	**Blue** „	**Brown** or **Colourless**
Iron.	**Brownish-yellow** „	**Bottle-green** „
Manganese.	**Purple** or **Pink** „	**Colourless** „
Nickel.	**Brownish-yellow** „	**Dusky-grey**‡ „

169. Moisten the substance with hydrochloric acid, and expose, on the end of a platinum wire (fig. 50) to the reducing flame.§ Again moisten with hydrochloric acid, and heat a second time, to be quite sure of the result.

Barium	**Green** flame (114).
Calcium (lime)	**Red** flame.
Copper	**Bluish-green** flame.
Potassium	**Violet** flame (149).
Sodium	**Yellow** flame.‖
Strontium	**Carmine** flame.

170. Heat the substance in a small tube sealed at one

† In exact blowpipe analysis, it is advisable, after fusion with carbonate of soda on charcoal, to grind the fused mass and surrounding charcoal to fine powder in an agate mortar, to suspend the powder in water, and pour off the lighter particles (*levigation*). On repeating the grinding and levigation several times, copper may be seen in red spangles, tin in brilliant white spangles, and iron, cobalt, and nickel as dark grey metallic powders, attractable by a magnetized knife-blade.

‡ Purple on adding a minute quantity of nitrate of potash.

§ Take care that the wire itself imparts no tinge to the flame. Should it do so, it must be cleaned by repeated moistening with hydrochloric acid and heating in the flame.

‖ *See* note to (154).

end (fig. 47), first in the lower part of the ordinary flame, and afterwards with the blowpipe.

(*a*) Is any *water* condensed on the cool part of the tube?

Is the water **acid** or **alkaline** to test-papers?

If acid (reddening blue litmus paper), the presence of *sulphuric, hydrochloric, nitric, acetic,* or *oxalic* acid may be suspected.

If alkaline (turning red litmus blue, or turmeric brown), *ammonia,* or *nitrogenized organic matter,* is probably present (109).

(*b*) Is there any **sublimate** deposited on the upper part of the tube?

This may be due to the presence of *ammonia* (109), of *arsenic* (111), of *mercury* (140), or of *sulphur* (156).

(*c*) Does the substance **carbonize** and emit any peculiar odour?

Indicating the presence of *organic matter,* either nitrogenized (145), or non-nitrogenized (119).

171. Mix the substance with about an equal quantity of bisulphate of potash, and heat in a small tube sealed at one end (fig. 47), in the lower part of the ordinary flame.

Brown acid vapours indicate *nitric acid.*

Violet vapours indicate *iodine.*

Yellow bleaching vapours indicate *chlorine,* either as a chloride (124), a hypochlorite, or a chlorate (121, 122).

Pungent acid vapours fuming in moist air indicate chlorine as a *chloride,* or fluorine as a *fluoride* (131).

Effervescence, without any particular odour, indicates *carbonic acid.*

172. Mix a very little of the powdered substance with dry carbonate of soda and charcoal, and heat in a small tube sealed at one end (fig. 47), first in the lower part of the ordinary flame, and afterwards, if necessary, with the blowpipe; to detect

Ammonium, by the **pungent odour** (109).

Arsenic, by the **shining black sublimate** (113).

Mercury, by the **grey sublimate of minute globules** (140).

173. Test for *silica* and for *manganese* by fusing on platinum wire with carbonate of soda (138, 150).

174. Test for *boracic acid* by fusing on platinum wire with fluor spar and bisulphate of potash (117).

175. Test for *phosphoric acid* by fusing on charcoal with boracic acid and iron wire (148).

Note.—It is often difficult to obtain good pieces of charcoal. Fragments of the bowls of tobacco-pipes may be used as supports, the substance to be tested being mixed with bitartrate of potash, which will serve both as flux and reducing agent. These fragments will also serve, instead of platinum wire in the borax-glass test (168).

The coloured flame test (169) is greatly enhanced in value by examining the flame with the *spectroscope*, but the description of this instrument exceeds the scope of this little work.

CHAPTER VI.

SPECIFIC GRAVITY.

176. By *specific gravity* is meant the relation between the weights of equal bulks of various kinds of matter. It has been found convenient to compare the specific gravities of all solids and liquids with that of water, which is reckoned as 1·000 or 1000. The specific gravity of substances heavier than water is consequently represented by a higher number, and of those which are lighter by a lower number than 1·000: that of lead, for instance, which is more than eleven times heavier than water, is represented by the number 11·44; while that of ether, which is considerably lighter than water, is represented by the number 0·724.

SECTION I.

Specific gravity of solids heavier than water.

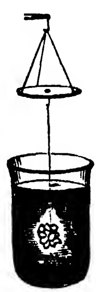


Fig. 53.

177. When the substance is solid and insoluble in water, its specific gravity may be ascertained in the following manner:—Suspend it by means of a horse-hair, from a hook attached to the scale-pan, making a small loop at one end of the hair, passing the other end through it, and enclosing the substance in the noose. Thus suspended, weigh it exactly. Then immerse it in water and weigh it again (fig. 53), care being

taken that it is covered on all sides by at least half an inch of water. Small bubbles of air frequently adhere to the surface, and these must be brushed off with a feather or camel-hair pencil, as they would tend to buoy it up, and cause the specific gravity to appear too low.

The results may be noted down as follows:—

Weight of the substance in air	=	
" " in water . . .	=	
Loss	=	

which number represents the weight of an equal bulk of water. Then by dividing the weight in air by the loss, or the weight of an equal bulk of water, the specific gravity is ascertained.

$$\frac{\text{Weight in air}}{\text{Loss}} = \text{Specific gravity.}$$

178. In this way determine the specific gravity of some of the following substances:—marble, amber, iron pyrites, sulphate of baryta, jet, lead, zinc, glass, and agate.*

SECTION II.

Specific gravity of solids lighter than water.

179. If the solid be lighter than water, as cork, a slight modification of the above process is necessary.

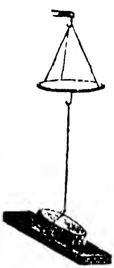


Fig. 54.

Weigh the substance first in air as before: then select a piece of lead of sufficient size to sink the light body in water when attached to it, and weigh it (the lead) in water, suspending it my means of a hair loop. If now the light substance be enclosed in the same loop with the lead (fig. 54), and immersed in water, it will be found that they will together weigh less than the lead did alone, owing to the buoyancy of the lighter body; and this difference, when added to the weight of the body in air, is equal to the weight of a corresponding bulk of water.

* The following are the specific gravities of these substances, some of which, however, vary considerably. Marble, 2·70; amber, 1·08; iron-pyrites, 4·9); sulphate of baryta, 4·47; jet, 1·30; lead, 11·44; zinc, 7·00; flint glass, 3·30; and agate, 2·60.

The results may be thus recorded:—

Weight of body in air	=	————
Weight of lead alone in water	=	
Weight of lead with body attached, in water	=	————
Difference	=	
Add weight of body in air	=	————
Weight of an equal bulk of water*	=	====

Having thus obtained the weight of the body in air, and the weight of an equal bulk of water, the specific gravity is calculated as before.

$$\frac{\text{Weight in air}}{\text{Weight of equal bulk of water}} = \text{Specific gravity.}$$

In this way ascertain the specific gravity of wood, cork, and charcoal.†

SECTION III.

Specific gravity of insoluble powders.

180. When the substance, whose specific gravity we wish to determine, is in the form of powder, or even small lumps, it is clear that some other method must be adopted than those just described. The following is the most simple, and, for common purposes, sufficiently accurate. Counterpoise‡ a small bottle furnished with a stopper; then fill it about two-thirds full of the powder to be examined, and again weigh; fill the bottle cautiously with water, care being taken that all air bubbles are expelled, and that none of the powder is washed out. Again weigh. Lastly, rinse the bottle thoroughly, fill it with distilled water, and weigh again.

* The principle of this calculation may not be evident at first sight. Let W represent the weight of the lead alone in water, and w the weight of the lead and cork in water; then w—W would be the weight of the cork alone in water; but, the cork being lighter than water, W is greater than w, and hence w—W is a negative quantity, and represents the buoyancy of the cork,—*i.e.* the excess of weight of an equal volume of water over that of the cork. By adding then to this difference the weight of the cork in air, we obtain the weight of an equal bulk of water.

† The specific gravity of these substances varies considerably, according to the degree of porosity: the following may be considered as the usual average: wood (beech), 0·85; cork, 0·14; and charcoal, 0·2 to 0·5.

‡ This is done by putting shots or strips of lead in a pill-box placed in the opposite scale, until their weight is equal to that of the bottle The bottle is placed in the scale-pan in the ordinary way. Bottles may be purchased which are made to contain exactly 1000 grains of distilled water.

From the data thus obtained, the specific gravity may be calculated as follows :—

Weight of the powder and water	=
Weight of the powder alone	=
Difference = weight of water left in the bottle.	=
Weight of water which fills the bottle . . .	=
Water left in the bottle after the powder was added	=
Weight of water displaced by, and equal in bulk to, the powder	=

Then as before :—

$$\frac{\text{Weight of the powder}}{\text{Weight of water displaced}} = \text{Specific gravity.}$$

In this way ascertain the specific gravity of sand, pounded glass, and shot.*

SECTION IV.

Specific gravity of liquids.

181. With a bottle similar to that used in the last experiment, the specific gravity of liquids may be readily determined. As the space occupied by a given weight of liquid varies with the temperature, or in other words, as the weight of a given volume of any liquid is greater or less as the temperature is lower or higher, it is necessary to observe that the temperature of the liquid during the experiment does not vary much from 60° F., which is usually taken as the standard. For the same reason the bottle should not be held in the warm hand during the experiment, as otherwise the heat would cause the liquid to expand, and become specifically lighter.

Fig. 55. Specific Gravity Glass.

Counterpoise the bottle, and weigh it full of distilled water: then, by filling it successively with other liquids, weighing, and comparing the different weights with that of water, the volume of liquid being always the

* The specific gravity of sand is about 2·60; flint glass, 3·30; and shot, 11·35.

same, the specific gravity is obtained by proportion, thus :—

Weight of water : 1·000 :: Weight of liquid : Specific gravity.

Care must be taken to clean the bottle thoroughly after each experiment, by washing it first with distilled water, and then with a little of the liquid whose density is to be ascertained.

Fig. 56.

Some of the following may be taken for practice: saturated solution of chloride of sodium, sulphuric acid, nitric acid, hydrochloric acid, solution of ammonia, solution of potash.*

182. The specific gravity of liquids may also be determined by another process, which, though not capable of so much accuracy as the last, is frequently useful when a specific-gravity bottle is not at hand.

Take a piece of solid glass rod, about the size of the figure (fig. 55), with one end drawn out and turned in the blowpipe flame. Weigh it first in the air and then in water, suspending it with a hair-loop (fig. 56). Then, having wiped it dry between each experiment, weigh it successively in the liquids, the specific gravities of which are to be determined. The difference between the weight of the glass in air and in the liquid, representing in each case the weight of a volume of the liquid equal to that of the glass; and the weight of a similar volume of water being known, the specific gravity may be ascertained by simple calculation.

Thus :

Weight of glass in air .
Weight of glass in liquid
Loss . . .

which is the weight of an equal volume of the liquid.

Then by proportion,

$$\left.\begin{matrix}\text{Weight of equal}\\ \text{volume of water}\end{matrix}\right\} : 1{\cdot}000 :: \left\{\begin{matrix}\text{Weight of equal}\\ \text{volume of liquid}\end{matrix}\right\} : \left\{\begin{matrix}\text{Specific gravity}\\ \text{of the liquid.}\end{matrix}\right\}$$

Determine in this way the specific gravities of some of the solutions already mentioned, and compare the results with those obtained with the specific-gravity bottle.

* The specific gravity of a saturated solution of chloride of sodium is 1·205. That of the other liquids will be found in the tables given in the Appendix.

CHAPTER VII.

HEATING SUBSTANCES IN GASES.

SECTION I.

Reduction of metallic oxides by hydrogen

183. A large number of the metallic oxides are decomposed and reduced to the metallic state when heated in an atmosphere of hydrogen gas; and from the facility with which the operation may be performed, and the accurate results which it gives when carefully conducted, it is frequently employed in estimating the quantity of oxygen present in oxidized compounds.

184. The apparatus which is required for the purpose is shown in the figure. (Fig. 57.) The bottle *a* is charged

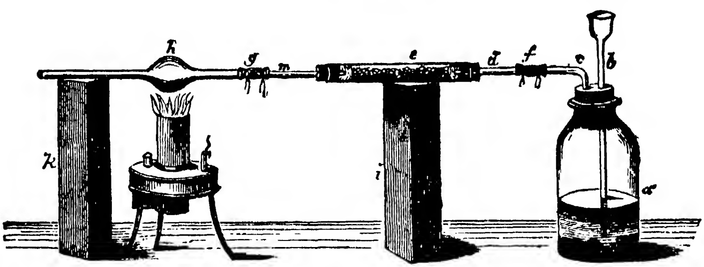


Fig. 57. Reduction of Oxide of Copper.

with zinc and dilute sulphuric acid to generate the hydrogen, which is dried while passing over fragments of chloride of calcium in the tube *e*; the gas then passes into the bulb-tube* *h*, which contains the oxide to be reduced, the bulb being heated by the lamp placed beneath.

* A piece of plain hard glass tubing (combustion tubing), about half an inch in diameter, provided with a cork and small tube at one end, and drawn out to a point at the other, may be substituted for the bulb-tube, provided that care be taken to introduce the powder into its centre, as may be easily done by placing it in a gutter of stiff paper, so as to occupy only about an inch, then sliding the gutter into the tube, and turning this half round upon its axis, so that the powder may fall from the paper on to the glass; the gutter is then carefully withdrawn, without disturbing the powder.

185. Take a piece of tubing *e*, about eight or ten inches long, and half an inch internal diameter, and having slightly fused the cut edges in the blowpipe flame, adapt a cork to each end: then, with a cork-borer or round file, perforate the corks so as to receive the small tubes *d* and *m*. In using the cork-borers, bore half-way through the centre of the cork, then reverse it, and bore in the opposite direction; the two holes meeting in the centre will be much smoother than if bored directly through. When the tube is of such a diameter as cannot be exactly matched by any of the cork-borers in the set, the hole should be bored by a smaller one, and afterwards enlarged by means of a round file, until it is of sufficient calibre to admit the tube, which must always fit perfectly tight. Remove one of the corks from the large tube, and push down to the other end a small loose bit of tow or cotton-wool, and nearly fill it with fragments of chloride of calcium; put in another bit of tow (the use of which is to prevent any of the smaller fragments falling out), and again fix the cork and small tube.

Next adapt a cork to the bottle, which should have a tolerably wide neck, and bore in it two holes to fit the tubes *b* and *c*, which pass through it, the former reaching nearly to the bottom of the bottle, the latter passing only just through the cork.* Put 300 grains of granulated zinc into the bottle, and fix the cork containing the tubes *b* and *c*.

186. In order to connect the different parts of the apparatus together, short pieces of vulcanized india-rubber tubing are employed; or, if a plain tube be substituted for the bulb-tube, the tube *c* may be directly inserted into the cork of the chloride of calcium tube, and the tube *m* into that of the plain tube.

187. Weigh the bulb-tube accurately, and place in the bulb 20 or 30 grains of oxide of copper previously well dried by a moderate heat; again weigh, to ascertain the weight of oxide operated on, and connect the apparatus as shown in the figure. The apparatus being thus arranged, cover the fragments of zinc with about an ounce of water, and add dilute sulphuric acid, by the funnel-tube, till a moderate effervescence takes place; when the gas

* When a funnel-tube is not at hand, a piece of wide tubing may be passed through the cork, and an ordinary funnel placed in it.

has been coming over about five minutes, apply a gentle heat to the bulb with a Bunsen burner or a spirit-lamp, and gradually increase it as long as any water is formed by the combination of the hydrogen with the oxygen of the oxide of copper. $CuO+H_2=Cu+H_2O$.

It is necessary to observe the precaution of not applying the heat immediately, since the apparatus at first contains an explosive mixture of hydrogen and common air, which would, if heat were applied, be in great danger of exploding, and seriously injuring the operator; by allowing five minutes to elapse, however, the whole of the common air is expelled, and the bulb may be heated without danger.

It is advisable to heat the anterior portion of the tube before applying any heat to the oxide of copper, so as to prevent the condensation of any large quantity of moisture, which is apt to crack the tube. A slight inclination towards the open extremity should be given to the bulb-tube, so that any drops of water which may condense shall have no chance of running back upon the hot part of the tube.

188. When the decomposition appears to be complete, no more water being produced,* expel by heat any moisture that may have condensed in the cool end of the tube, remove the lamp, and allow the bulb-tube to cool: then disconnect the apparatus, and weigh the bulb containing the reduced metallic copper, the loss of weight indicating the quantity of oxygen which has been removed. Ascertain by calculation the proportion of oxygen in 100 parts of the oxide, and compare the experimental result with what is theoretically correct.

SECTION II.

Heating substances in an atmosphere of carbonic acid.

189. It is sometimes required in analysis to separate two substances, one of which is volatile at a high temperature, and the other fixed, so that by merely heating the

* This is known by holding a piece of cold glass close to the opening at the end of the tube, and observing whether any moisture is condensed upon its surface; if not, it may be inferred that no water is coming off. Should the hydrogen burn at the end of the tube, it must be extinguished before applying this test, by holding a thick piece of iron in the flame, and blowing it.

mixture, and weighing before and afterwards, the weight of each ingredient is determined. In some cases, however, it happens that the non-volatile body, when heated in atmospheric air, combines with oxygen, forming a volatile compound; so that here it is necessary to conduct the operation in an atmosphere of some gas incapable of combining with it, as hydrogen or carbonic acid. For instance, in the analysis of gunpowder, which consists of a mixture of nitrate of potash, sulphur, and charcoal, the nitrate of potash is first dissolved out with water, and the insoluble residue, consisting of sulphur and charcoal, is heated in a current of hydrogen or carbonic acid, when the sulphur, being volatile, is expelled; whereas, if the mixture were to be heated in common atmospheric air, the carbon as well as the sulphur would disappear, since it would combine with oxygen, and become converted into carbonic acid, which is a gas.

190. The apparatus required for this purpose is the same as that used for the reduction of metallic oxides by hydrogen (184). Fill the generating bottle *a* about one-third full of water, and put in some fragments of marble; when the apparatus is arranged, pour in from time to time a little hydrochloric acid through the tube *b*, so as to maintain a moderate effervescence. Weigh the bulb-tube, and put into it about ten grains of the mixture of sulphur and charcoal; weigh a second time, to ascertain how much is used in the experiment, and connect the apparatus together. Allow the gas to come over for about five minutes, in order to displace the common air (which might otherwise cause the loss of some of the charcoal, by conversion into carbonic acid), and then heat the mixture as long as any sulphur is volatilized, taking care to chase out any sulphur which may have condensed on the cool part of the tube. As soon as the apparatus is cold, weigh the bulb-tube again, when the loss of weight will represent the quantity of sulphur contained in the mixture. The percentage of sulphur is then ascertained by calculation.*

Weight of mixture : loss of weight :: 100 : percentage of sulphur.

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* The glass tube must be very gently heated, for, at a red heat, the carbonic acid gas would convert part of the carbon into carbonic oxide gas. The residue left when gunpowder is exhausted with water contains 40 per cent. of sulphur.

PART II.

RECOGNITION OF METALS AND NON-METALLIC BODIES IN THEIR MOST FREQUENT FORMS OF COMBINATION.

CHAPTER I.

SECTION I.

Introductory.

191. QUALITATIVE analysis has for its object the determination of the elements or compounds which are contained in any given substance; and those elements and compounds are recognised by certain characteristic appearances which they present when exposed to the action of tests or reagents, or when otherwise treated, as when submitted to heat, &c.

Before proceeding, therefore, to the more complicated processes of analysis, it is advisable that the student should make himself familiar with the action of reagents on the compounds most commonly met with in such investigations, in order to enable him properly to interpret the language in which Nature, through his experiments, replies to his inquiries.

With this purpose in view, he should not merely apply his tests, and superficially note whether a precipitate is or is not formed, but he should endeavour to impress on his recollection the exact appearance which it presents, both as to colour and also as to physical structure; whether it is crystalline, curdy, or gelatinous; whether it separates immediately from the solution, or requires time for its development; as well as the action of solvents (as acids

and alkalies) upon it. Besides the increased facility which he will thus gain in making subsequent experiments, he will be acquiring habits of close and accurate observation, which will be of infinite value to him, not only in pursuing the study of chemistry, but in almost every occupation of life.

SECTION II.

Analytical Classification.

192. In describing the action of reagents, and the rudiments of chemical analysis, all the rarer substances will be omitted, as they would only tend to confuse the student.* The following are those which will be treated of, as being most commonly met with in analysis. The metals are classified according to their behaviour with *hydrosulphuric acid*, *hydrosulphate of ammonia*, and *carbonate of ammonia*. Those in Class I. are precipitated as sulphides from acidified solutions by hydrosulphuric acid: those in Class II. are not affected by hydrosulphuric acid when an excess of hydrochloric acid is present, but are thrown down either as sulphides or oxides when their neutral solutions are treated with hydrosulphate of ammonia: those in Class III. are not precipitated by either hydrosulphuric acid or hydrosulphate of ammonia, but are thrown down as carbonates, by carbonate of ammonia: and those in Class IV. are unaffected by any of those reagents.†

METALS AND THEIR PRINCIPAL OXIDES.

Class I.

Metals.	*Oxides.*
Antimony, Sb	Oxide of Antimony, Sb_2O_3.
Arsenic, As	Arsenious Acid, As_2O_3.
	Arsenic Acid, As_2O_5.
Bismuth, Bi	Oxide of Bismuth, Bi_2O_3.
Copper, Cu	Oxide of Copper, CuO.
Lead, Pb	Oxide of Lead, PbO.
Mercury, Hg	Mercurous Oxide, Hg_2O.
	Mercuric Oxide, HgO.
Silver, Ag.	Oxide of Silver, Ag_2O.
Tin, Sn	Stannous Oxide, SnO.
	Stannic Acid, SnO_2.

* In the Appendix will be found a table showing the behaviour of most of the rarer substances with reagents.

† See table of Analytical Classification in the Appendix.

Class II.

Metals.	*Oxides.*
Aluminium, Al	Alumina, Al_2O_3.
Chromium, Cr	Oxide of Chromium, Cr_2O_3.
	Chromic Acid, CrO_3.
Cobalt, Co	Oxide of Cobalt, CoO.
Iron, Fe	Ferrous Oxide, FeO.
	Ferric Oxide, Fe_2O_3.
Manganese, Mn	Oxide of Manganese, MnO.
	Binoxide, MnO_2.
	Manganic Acid, H_2MnO_4 (?)*.
	Permanganic Acid, $HMnO_4$ (?).
Nickel, Ni	Oxide of Nickel, NiO.
Zinc, Zn	Oxide of Zinc, ZnO.

Class III.

Barium, Ba	Baryta, BaO.
Calcium, Ca	Lime, CaO.
Magnesium, Mg	Magnesia, MgO.
Strontium, Sr	Strontia, SrO.

Class IV.

Ammonium (hypothetical) .	NH_4.
Potassium, K	Potash, K_2O.
Sodium, Na	Soda, Na_2O.

NON-METALLIC BODIES AND THEIR PRINCIPAL ACIDS.

Non-metallic bodies.	*Acids.*
Nitrogen, N	Nitric Acid, HNO_3.
Sulphur, S	Hydrosulphuric Acid, H_2S.
	Sulphurous Acid, H_2SO_3.
	Hyposulphurous Acid, $H_2S_2O_3$ (?).
	Sulphuric Acid, H_2SO_4.
Phosphorus, P	Phosphoric Acid, H_3PO_4.
Carbon, C	Carbonic Acid, CO_2.
	Oxalic Acid, $H_2C_2O_4$.
	Hydrocyanic Acid, HCN.
	Hydroferrocyanic Acid, $H_4C_6N_6Fe$.
	Tartaric Acid, $H_2C_4H_4O_6$.
	Citric Acid, $H_3C_6H_5O_7$.
	Gallic Acid, $H_3C_7H_3O_5$.
	Tannic Acid, $C_{14}H_{10}O_9$ (?).
	Benzoic Acid, $HC_7H_5O_2$.
	Acetic Acid, $HC_2H_3O_2$.
Boron, B	Boracic Acid, H_3BO_3.
Silicon, Si	Silicic Acid, SiO_2.
Chlorine, Cl	Hydrochloric Acid, HCl.
	Hypochlorous Acid, HClO (?).
	Chloric Acid, $HClO_3$.
Iodine, I	Hydriodic Acid, HI.
Fluorine, F	Hydrofluoric Acid, HF.

193. Should the student find that the action of any test

* The note of interrogation implies that the formula is not satisfactorily established by analysis.

does not agree with that described, it may be owing to some impurity contained in the test-liquid, in which case he may examine it in the manner described in the section on reagents.

It will be observed that, in general, only those reactions are set down which admit of practical application in the systematic course of analysis, those which possess merely a general interest being omitted.

The student is cautioned against supposing that any reaction which is ascribed to one substance, and not to others of the same group, may serve as a characteristic test for that substance, since such an application will always receive a special notice, and the reaction is omitted in the case of the other substances, merely because it is not applied in the systematic course of analysis.

CHAPTER II.

METALS BELONGING TO CLASS I.

Silver, Lead, Mercury, Bismuth, Copper, Tin, Antimony, Arsenic.

194. These metals are distinguished from those of the succeeding classes, by being precipitated from their acidified solutions when treated with hydrosulphuric acid. It is remarkable that nearly all the metals, the compounds of which are most eminently poisonous, belong to this class.

SECTION I.

Silver (Ag).

A solution of nitrate of silver ($AgNO_3$) may be used.

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195. Pour a few drops of solution of $AgNO_3$ into a test-tube, and add some hydrosulphuric acid.* A black

* Always smell hydrosulphuric acid before using it, since, when kept, its hydrogen becomes oxidised by the air, and sulphur deposited. If it be fit for use, it will smell very strongly of the gas.

precipitate of sulphide of silver will be produced, which will not dissolve on adding a few drops of dilute nitric acid. $2AgNO_3+H_2S=2HNO_3+Ag_2S$.

196. (C)* *Hydrochloric acid* produces in solutions of silver a white curdy precipitate of chloride of silver, which is readily soluble in ammonia.

$$AgNO_3+HCl=HNO_3+AgCl.$$

If the ammoniacal solution be neutralized with nitric acid, the chloride is reprecipitated. Compare (199, 208).

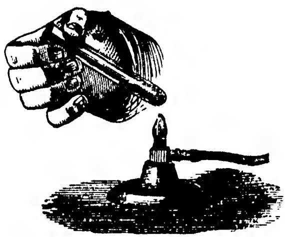

Fig. 58.

197. *Identification of metallic silver.*—Dissolve a scrap of silver in dilute nitric acid, with the aid of heat (fig. 58), and ascertain the presence of the metal by adding hydrochloric acid, and dissolving the precipitate in ammonia.

[For the blowpipe test, *see* (152).]

SECTION II.

Lead (Pb).

A solution of the acetate or the nitrate of lead $Pb(NO_3)_2$ may be used.

---

198. (C) *Hydrosulphuric acid* throws down, in solutions containing lead, a dense black precipitate of sulphide of lead.† $Pb(NO_3)_2+H_2S=2HNO_3+PbS$.

If the sulphide be boiled with dilute nitric acid, a little of it is converted into the insoluble sulphate of lead, but the greater part is dissolved.

199. (C) *Hydrochloric acid* throws down a white and often crystalline precipitate of chloride of lead. If the solution with the precipitate be boiled, a portion of the

* Tests marked (C) are those generally employed in the analytical course, and should therefore receive particular attention.

† In solutions of lead to which an excess of hydrochloric acid has been added (199), hydrosulphuric acid often produces a red precipitate composed of sulphide and chloride of lead, which becomes black after a time, or on boiling, in contact with an excess of hydrosulphuric acid.

chloride dissolves, and is deposited again on cooling, in the form of needle-shaped crystals. If the solution of lead is dilute, the chloride does not precipitate, as it is somewhat soluble in water.

$$Pb(NO_3)_2+2HCl=PbCl_2+2HNO_3.$$

The chloride of lead is not dissolved by an excess of ammonia. Compare (196).

200. (C) *Dilute sulphuric acid* produces a white granular precipitate of sulphate of lead, which is sparingly soluble in acids, but soluble in acetate of ammonia mixed with excess of ammonia* on boiling.

$$Pb(NO_3)_2+H_2SO_4=PbSO_4+2HNO_3.$$

If the precipitate be moistened with a solution of ammonium sulphide, it is instantly blackened, owing to the formation of sulphide of lead: it is distinguished in this way from the insoluble sulphates of barium and strontium.

201. (C) *Chromate of potash* gives a fine yellow precipitate of lead chromate which is insoluble in dilute acids. This substance is the basis of the pigment known in commerce as chrome yellow.

$$K_2CrO_4+Pb(NO_3)_2=2KNO_3+PbCrO_4.$$

202. (C) *Iodide of potassium* also gives a beautiful yellow precipitate of lead iodide, which is rather lighter in tint than the chromate. If the iodide thus formed be boiled with water, it dissolves, especially if a little hydrochloric acid be added, and again separates on cooling, in the form of brilliant crystalline scales, which are extremely beautiful. $2KI+Pb(NO_3)_2=2KNO_3+PbI_2$.

203. *Identification of metallic lead.*—Boil a small piece of lead (*e.g.* a shot) with a little dilute nitric acid, which will gradually dissolve it; test different portions of the solution with dilute sulphuric acid (200), and hydrochloric acid (199). Strong nitric acid will not easily dissolve lead, the metal becoming coated with nitrate of lead, which is not soluble in the strong acid.

[For the blowpipe test, *see* (134).]

* Acetate of ammonia may be made by gradually adding acetic acid to solution of ammonia, till the liquid is slightly acid to blue litmus paper.

SECTION III.

Mercury (Hg).

A. *As mercurous oxide** (Hg_2O) *or some corresponding compound.*

Fig. 59.

204.† (C) Heat a very little calomel Hg_2Cl_2 in a small tube sealed at one end (*not a test tube*). It becomes pale yellow, and, being volatile, it sublimes, and condenses in the upper part of the tube; on cooling, the colour disappears.

205. (C) Mix a little dry carbonate of soda with a very minute quantity of calomel, and put the mixture into a tube, *a* (fig. 59); then cover it with a layer of carbonate of soda in powder, about a quarter of an inch deep, *b*, and apply heat (140). The calomel is decomposed, and minute globules of metallic mercury condense in the cool part of the tube at *c*. $Hg_2Cl_2+Na_2CO_3=Hg_2+2NaCl+CO_2+O$.

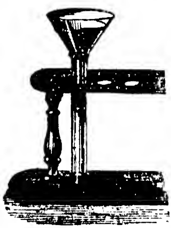

Fig. 60. Filtering into Tube.

206. Boil a little calomel with distilled water in a test-tube; filter the water into another tube (fig. 60), and test it with ammonium sulphide; no effect is produced, proving that the calomel is insoluble in water.‡

207. (C) *Potash* or *ammonia* poured on the calomel, decomposes it, turning it black, owing to the formation of the black oxide of mercury.

$$Hg_2Cl_2+2KHO=Hg_2O+2KCl+H_2O.$$

208. (C) *Hydrochloric acid*, added to a solution of a mercurous salt (*e.g.* the protonitrate), throws down a white precipitate of mercurous chloride (calomel).

$$2HgNO_3 + 2HCl = 2HNO_3 + Hg_2Cl_2.$$

Ammonia converts the precipitate into a grey compound of mercury. Compare (196, 199).

$$Hg_2Cl_2+2NH_3=NH_2Hg_2Cl+NH_4Cl.$$

* Also called protoxide or suboxide of mercury.

† The blowpipe tests for calomel are repeated here, on account of its special importance.

‡ If the calomel contained any corrosive sublimate, the sulphide would produce a black precipitate.

209. (C) Place a strip of clean *copper* in the mercurial solution, and observe the deposition of metallic mercury.

B. *As Mercuric Oxide* (HgO) *or some corresponding compound.*

The mercuric chloride ($HgCl_2$) (bichloride of mercury or corrosive sublimate), either solid or in solution may be used.

210. Heat a small fragment of $HgCl_2$ in a small tube sealed at one end; it fuses, boils, and sublimes into the upper part of the tube (141).

211. (C) Repeat the experiment described in (205), using a very small particle of $HgCl_2$ instead of calomel; metallic mercury sublimes in both cases.

212. Boil a little with water, in which it readily dissolves, thus differing from mercurous chloride.

213. (C) Test a solution of $HgCl_2$ with a small quantity of *hydrosulphuric acid.* A white precipitate is first formed, which, on the addition of more of the precipitant, gradually becomes darker, and ultimately black. This change of colour is owing to the formation of a double compound of HgS and $HgCl_2$, which is white; and when the hydrosulphuric acid is added in excess, the whole of the mercury is converted into the mercuric sulphide (HgS).* The precipitate is insoluble in hydrochloric and nitric acids separately, but is readily dissolved by a mixture of the two on heating, and again converted into $HgCl_2$.

214. *Ammonia* throws down *white precipitate.*

$$HgCl_2+2NH_3=NH_2HgCl+NH_4Cl.$$

215. (C) *Potash* gives a yellow precipitate of mercuric oxide. $HgCl_2+2KHO=HgO+2KCl+H_2O$.

216. (C) When *protochloride of tin* is added in small quantity, the mercuric chloride is reduced to the state of mercurous chloride, which separates as a white precipitate. If the salt of tin be added in excess the mercury is reduced to the metallic state. $HgCl_2+SnCl_2=Hg+SnCl_4$.

217. (C) *Potassium iodide* causes a most beautiful red

* $HgCl_2 + H_2S = 2HCl + HgS$.

precipitate of mercuric iodide, which surpasses even vermilion in brilliancy of colour. $HgCl_2+2KI=HgI_2+2KCl$. It is readily soluble in an excess of either of the solutions, especially in the iodide of potassium. Compare (202).

218. (C) If a solution containing mercury be acidified with hydrochloric acid, and boiled with a few slips of clean copper, the surface of the latter becomes coated with a lustrous film of metallic mercury, and if the slips be dried upon blotting-paper and heated in a small tube closed at one end (not a test-tube), globules of mercury may be obtained. $HgCl_2+Cu=CuCl_2+Hg$.

SECTION IV.

Bismuth (Bi).

A solution of the chloride ($BiCl_3$) may be used.

---

219. (C) Mix a concentrated solution of the chloride with a considerable quantity of *water*, which causes a white precipitate of oxychloride of bismuth.

$$3BiCl_3+3H_2O=BiCl_3Bi_2O_3+6HCl.$$

A similar decomposition takes place when solutions of many of the soluble salts of bismuth are diluted with much water. The precipitates thus formed are usually distinguishable from those produced under the same circumstances in solutions of antimony, by being insoluble in tartaric acid.

220. (C) *Hydrosulphuric acid* and *ammonium sulphide* throw down, from solutions of bismuth, a black precipitate of bismuth sulphide, which is insoluble in cold dilute acids, but soluble in hot nitric acid.

$$2BiCl_3+3H_2S=Bi_2S_3+6HCl.$$

221. (C) *Ammonia* gives a white precipitate of bismuth hydrate, which is insoluble in an excess of the precipitant.* If the precipitate be redissolved in as little hydrochloric acid as possible, the solution will become milky when mixed with a large quantity of water (219).

222. (C) *Iodide of potassium* gives, in strong solutions,

* $BiCl_3+3\ NH_4HO=BiH_3O_3+3\ NH_4Cl$. (?)

a brown precipitate of bismuth iodide; in dilute solutions, only a red brown colour. *One drop* of *acetate of lead* added to the red brown solution gives a brown precipitate composed of lead and bismuth iodides; if this precipitate be dissolved by boiling with water and a little hydrochloric or acetic acid, it will crystallize out, with a very beautiful colour and appearance, on cooling the test-tube in a stream of water. Compare (202, 217).

222*a*. Add to the solution some $SnCl_2$ (prepared by dissolving a piece of tin in conc. HCl) and excess of potash. A black precipitate of bismuthous oxide (BiO) is obtained.

[For the blowpipe test, *see* (116).]

SECTION V.

Copper (Cu).

A solution of sulphate of copper ($CuSO_4$) may be used.

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223. (C) *Hydrosulphuric acid* and *ammonium sulphide* throw down a black precipitate of sulphide of copper from solutions of copper salts, whether neutral, acid, or alkaline.

$$CuSO_4+H_2S=CuS+H_2SO_4.$$

224. (C) *Ammonia*, when added in small quantity, throws down a pale blue precipitate, consisting of a basic salt of copper, which immediately redissolves when the ammonia is added in excess; the solution thus formed has a beautiful deep blue colour, owing to the formation of the ammonio-sulphate of copper ($CuSO_4.4NH_3.H_2O$).

225. *Potash* produces in cold solutions of copper a pale blue precipitate of cupric hydrate (CuH_2O_2). If the mixture be boiled, or if the potash be added to a hot solution, the precipitate becomes black, owing to the decomposition of the hydrate, and formation of the black oxide (CuO). The potash must for this purpose be added slightly in excess, as otherwise the precipitate would consist of a basic salt, which would not become black when boiled.

226. (C) *Potassium ferrocyanide* gives, even in very dilute solutions, a mahogany-coloured precipitate of cupric ferrocyanide (Cu_2Cy_6Fe), which is insoluble in dilute acids.

227. (C) A piece of clean *iron* (a knife-blade), when

placed in a solution containing copper, causes a precipitation of metallic copper on its surface.

This is an extremely delicate test, and by this means the whole of the copper may be removed from a liquid, especially if a slight excess of acid is present.

228. *Identification of metallic copper.*—Dissolve a very small piece of copper in dilute nitric acid (22); test one part of the solution with ammonia (224), and the other with ferrocyanide of potassium (226).

[For the blowpipe tests, *see* (128, 129, 130).]

SECTION VI.

Tin (Sn).

A. *As protoxide* or some corresponding compound.*

A solution of protochloride of tin (stannous chloride $SnCl_2$) may be used.

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229. (C) *Hydrosulphuric acid* gives in solutions of stannous salts, either neutral or with excess of acid, a dark brown precipitate of stannous sulphide (SnS), which is soluble in ammonium sulphide containing an excess of sulphur (which is shown by the yellow colour). The solution is facilitated by heating.

If the solution thus formed be neutralized with hydrochloric acid, a yellow precipitate of stannic sulphide (SnS_2) is produced, which was formed by the action of the yellow ammonium sulphide upon the stannous sulphide.

$$SnS+(NH_4)_2S_2=(NH_4)_2S.SnS_2.$$

230. (C) *Mercuric chloride* produces, even in very dilute solutions, at first a white precipitate of mercurous chloride, which is often highly crystalline, and gives a silky appearance to the liquid. If a sufficient quantity of the tin salt be present, this white precipitate is converted after a time into grey metallic mercury (216).

* Also called *stannous oxide.*

B. *As peroxide of tin* or some corresponding compound.*

A solution of the perchloride (stannic chloride $SnCl_4$) may be used.

231. (C) *Hydrosulphuric acid* gives, in acid solutions, a pale yellow precipitate of stannic sulphide which is in some cases formed only on heating. It is dissolved when heated with ammonium sulphide. Compare (229).

232. *Ammonia* or *potash* throws down, from acid solutions, a bulky white precipitate of stannic hydrate (H_2SnO_3) which is soluble in an excess of the precipitant, especially when potash is used and heat applied, forming potassium stannate. K_2SnO_3.

The stannic hydrate when thus formed by precipitation with potash, is readily soluble both in potash and nitric acid, in which respect it differs from that formed by the action of nitric acid on metallic tin, though both yield the same results when analysed.

233. (C) *Carbonate of ammonia*, in acid solutions, produces a white precipitate of stannic hydrate, which is not dissolved on boiling with an excess of the carbonate. Compare (240).

234. If a piece of *zinc* be allowed to remain for a minute or two in a solution of tin, the latter will be deposited in the metallic state, and may be identified by boiling it in hydrochloric acid and testing with mercuric chloride (230).

235. (C) *Identification of metallic tin.*—Boil a small fragment of tin with dilute nitric acid; it is converted into a white insoluble powder, which is metastannic acid. Boil another portion of the metal with hydrochloric acid, which slowly dissolves it as stannous chloride; pour a little of the solution into another tube, and test with perchloride of mercury (230). Continue to boil the rest of the tin with the acid, and add dilute nitric acid very carefully at intervals, until the whole is dissolved. Test the stannic chloride thus produced with hydrosulphuric acid (231), and with carbonate of ammonia (233).

[For the blowpipe test, *see* (161).]

* Also called *stannic acid* and *binoxide of tin.*

SECTION VII.

Antimony (Sb).

236. Add *dilute hydrochloric acid*, drop by drop, to solution of tartar-emetic;* a white precipitate of oxide of antimony will be produced at first, but will be easily dissolved by the acid. Place a little of the solution on platinum foil, and dip in it a piece of zinc, touching the platinum. Antimony deposits as a black stain upon the platinum; rinse the latter, and place some yellow ammonium sulphide upon it; the stain will be dissolved, and orange antimonious sulphide will be left on evaporation.

237. (C) To this solution (containing only just enough acid to redissolve the precipitate) add eight or ten times its volume of *water*, when it will become milky from the separation of antimonious oxychloride ($SbCl_3.Sb_2O_3$), but may be rendered clear again by adding tartaric acid. Compare (219).

238. (C) Acidify a solution of tartar-emetic with hydrochloric acid, and add *hydrosulphuric acid*, which will produce an orange precipitate of antimonious sulphide. (Sb_2S_3).†

239. (C) Collect the antimonious sulphide upon a small filter, wash it once or twice with water, perforate the filter with a glass rod and wash the precipitate into a test-tube, using as little water as possible. Add carbonate of ammonia and heat gently; the sulphide dissolves only to a very slight extent, and is thus distinguished from arsenious sulphide. Add a little ammonium sulphide, and the precipitate will readily dissolve, but may be reprecipitated by dilute hydrochloric acid, though its colour will now be paler than before, because a little sulphur is precipitated together with it.

240. (C) Add *carbonate of ammonia* to solution of antimonious chloride. A white precipitate of antimonious hydrate is produced, which dissolves almost entirely on boiling with an excess of the carbonate of ammonia. Compare (233).

* Potassio-tartrate of antimony, derived from tartaric acid ($H_2C_4H_4O_6$) by the substitution of K and Sb for H_4. Tartar-emetic$=C_4H_2KSbO_6$.

† Antimonic acid would furnish a similar precipitate of antimonic sulphide (Sb_2S_5).

241. (C) *Identification of metallic antimony.*—Boil a little powdered antimony with dilute nitric acid, which will convert it into a white powder (antimonic acid—$HSbO_3$). Boil another portion of the metal with hydrochloric acid, and add nitric acid, very carefully, drop by drop, till the antimony is dissolved, as terchloride. Prove the presence of antimony in the solution by largely diluting one portion (237), and by testing another with hydrosulphuric acid (238).

[For the blowpipe test for antimony, *see* (110). The antimoniettod hydrogen test is described in the Section on Arsenic.]

SECTION VIII.

Arsenic (As).

A. *As Arsenious Acid* (As_2O_3).*

242. On account of the highly poisonous nature of arsenic, great care should be taken, in the following experiments, not to use more than is absolutely necessary to exhibit its peculiarities. In all these experiments (except those of solution), a fragment of the size of a small pin's head is quite sufficient. There is also another advantage in using such small quantities—namely, that in most medico-legal investigations, the quantity to be looked for is very minute, and it is consequently very important that the student should make himself familiar with the appearances which would, under these circumstances, present themselves.

243. † (C) If a small fragment of arsenious acid be heated on charcoal before the blowpipe, it is wholly volatilized, and a smell of garlic will be perceptible. Both metallic arsenic and arsenious acid are volatile when heated, but the fumes of the latter have no smell.

244. (C) Place a fragment of arsenious acid in a small tube sealed at one end (95) (not a test-tube), warm the upper part of the tube by drawing it once or twice through a gas or spirit flame, and apply a gentle heat to

* Commence this section by setting some arsenious acid to boil with water (*see* 247), and make the other experiments (242 to 246) in the interval.

† It has been thought advisable to repeat the blowpipe tests for arsenic (already described in Part I.), in order to exhibit in this section all the leading characters of that important poison.

the arsenious acid. It sublimes without decomposition, and condenses in the cool part of the tube, in the form of minute sparkling octahedral crystals (fig. 61), which should be examined with a lens, as they are highly characteristic. The size and regularity of the crystals depend on the slowness with which the vapour is condensed. If the surface of the glass on which the condensation takes place is quite cold, the sublimate is often amorphous.*

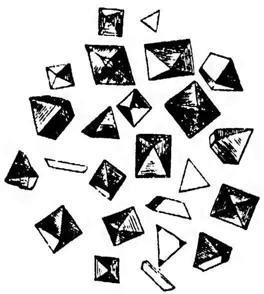

Fig. 61. Crystals of Arsenious Acid

245. (C) Mix a little arsenious acid with black flux (or a mixture of carbonate of soda and charcoal), which if at all damp should be previously dried (113), and heat a little of the mixture in a small tube, sealed at one end, before the blowpipe. The arsenious acid is deoxidized by the carbon of the flux, and the metallic arsenic thus reduced sublimes, and condenses in the upper part of the tube, forming a more or less brilliant metallic crust (*a*, fig. 49, p. 42). $2As_2O_3+C_3=3CO_2+As_4$.

If a moderate heat be now applied to the sublimate, it will again volatilize, and if any of the vapour escapes from the tube, it may be recognised by its characteristic odour of garlic.

246. (C) Cut off, by means of a file, the portion of the tube containing the crust, wrap it in a piece of paper, break it into fragments, and place some of them in another tube. Sublime the arsenic by a gentle heat in the tube, and observe the conversion of the metal into crys-

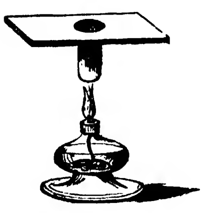

Fig. 62.

* Dr. Guy has devised a very simple method of obtaining this sublimate, and others of a similar kind, upon a flat slide for microscopical examination. In a flat plate of metal or porcelain, a hole is drilled, sufficiently large to receive a very short test-tube, about an inch and a half long, and half an inch in diameter (fig. 62) ; the arsenious acid is gently heated in this tube over a spirit-lamp flame, and the crystalline sublimate is allowed to deposit upon a glass slide slightly warmed and placed over the mouth of the tube. The binocular microscope permits the examination of sublimates in tubes.

talline arsenious acid, which is formed by the action of the atmospheric oxygen contained in the tube.

247. Boil a few grains of arsenious acid with water (in which it is sparingly soluble), in a flask: filter the solution from the undissolved portion, and retain it for testing.

248. (C) *Hydrosulphuric acid* added to a solution of arsenious acid in water, gives it a bright yellow colour, due to the formation of arsenious sulphide.

$$As_2O_3 + 3H_2S = As_2S_3 + 3H_2O.$$

If the solution be acidified with hydrochloric acid, a complete separation of the arsenic may be effected as bright yellow As_2S_3.

249. (C) Pour a little of a solution containing the yellow precipitate into a test-tube, and add a few drops of ammonia, which will very easily dissolve it. (Sulphide of cadmium, which much resembles the sulphide of arsenic, is insoluble in ammonia.)

250. (C) Collect the sulphide of arsenic upon a small filter (72), and wash it twice or thrice with water; pour over it a little warm solution of carbonate of ammonia, and observe that the sulphide of arsenic readily dissolves. Compare (239).

251. (C) Repeat the experiment with ammonium sulphide, which will dissolve it even more readily.

252. (C) Dry the remainder of the sulphide of arsenic very carefully,* mix a little of it with black flux, or with dried carbonate of soda and a little charcoal (113), and heat it in a tube; observe the formation of a metallic crust of arsenic, and identify it as in (246).

253. A very excellent method of testing the sulphide of arsenic was proposed by Fresenius: it consists in reducing the sulphide to the metallic state by fusion with sodium carbonate and potassium cyanide, and carrying the vapour of the metal, by means of a gentle stream of carbonic acid, into the narrow point of a glass tube, when

* A hot brick is very serviceable for drying precipitates on filters, especially where many students are working, since several bricks may always be kept upon the furnaces and carried to different parts of the laboratory as they are wanted. When the quantity of sulphide of arsenic is too minute to be removed from the filter, it may be dissolved off in a few drops of ammonia, the solution evaporated nearly to dryness, and a few drops of concentrated nitric acid added: on continuing the evaporation to dryness, arsenic acid will be left, which will assume a red-brown colour when moistened with a drop of nitrate of silver, especially if a trace of very dilute ammonia be added.

the smallest quantity becomes visible. Fig. 63 will show the arrangement adopted for this purpose. The larger

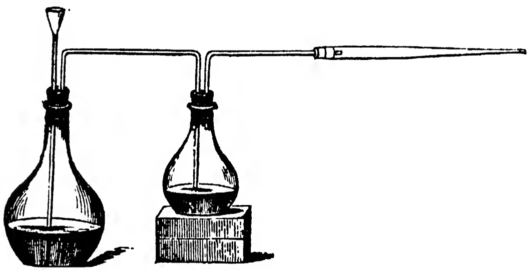

Fig. 63.

flask contains fragments of marble covered with water, to which hydrochloric acid is added through the funnel tube for the purpose of evolving carbonic acid, which is dried by passing through oil of vitriol in the smaller flask. The sulphide of arsenic is mixed with about 12 parts of a mixture of 3 parts of sodium carbonate and 1 part of potassium cyanide (previously well dried); the mixture is introduced into the tube* at *d* by means of a paper gutter

Fig. 64.

(note to 184), and the tube is then attached to the apparatus. As soon as all the air has been expelled, and the carbonic acid is passing through the oil of vitriol at the rate of about a bubble in a second, the mixture is thoroughly dried by gently heating the tube throughout its whole length with a spirit-lamp; when no more moisture is visible, the shoulder is so heated that no arsenic may condense there, and is kept hot while a second spirit-lamp is employed to heat the mixture; the heat may be ultimately increased by the blowpipe, if necessary; the metallic arsenic condenses in the narrow part of the tube, at *e*, and any of the tests may be applied in order to complete its identification.

* The tube is made of stout German glass.

Antimony gives no deposit when its sulphide is treated in this manner.

254. *Ammonium sulphide,* when added to a solution of arsenious acid, also causes the formation of the yellow sulphide, which, however, does not precipitate, but remains dissolved as ammonium sulpharsenite $(NH_4)_3AsS_3$. If hydrochloric acid be added in excess to the mixture, the sulphide of arsenic is immediately precipitated, of a somewhat lighter colour than that thrown down by hydrosulphuric acid, owing to the admixture of a little sulphur derived from the ammonium sulphide.

$$2(NH_4)_3AsS_3+6HCl=6NH_4Cl+3H_2S+As_2S_3.$$

255. *Ammonio-nitrate of silver** throws down, in solution of arsenious acid, a canary-coloured precipitate of silver arsenite (Ag_3AsO_3), which is soluble both in ammonia and nitric acid. It must be remembered that *phosphate of soda* also produces, with nitrate of silver, a similar precipitate, which is equally soluble in nitric acid and ammonia.

A solution of arsenious acid in water gives no precipitate with *nitrate of silver* unless ammonia is added, but this must be done very carefully, as a very slight excess will redissolve the yellow precipitate.

256. *Ammonio-sulphate of copper*† produces in solution of arsenious acid a delicate green precipitate of arsenite of copper, which dissolves readily both in ammonia and nitric acid. It must be borne in mind, in employing this test, that a similar precipitate is produced when the solution of copper is added to liquids containing some vegetable substances, though no arsenic may be present.

256*a*. *Stannous chloride* $(SnCl_2)$ gives a dark brown precipitate of As, when heat is applied.

Marsh's Test.

257. (C) It is well known that when zinc is treated with dilute sulphuric acid, hydrogen gas is given off. If, in addition to the zinc and dilute sulphuric acid, either of the oxides of arsenic be present, the arsenic combines with some of the hydrogen, and forms a gaseous com-

* Prepared by adding very dilute ammonia to nitrate of silver, drop by drop, till the precipitate at first produced is exactly redissolved.

† Prepared in a similar manner, with sulphate of copper.

pound called *arseniëtted hydrogen*, which passes off mixed with the excess of hydrogen.*

$$As_2O_3+Zn_6+6H_2SO_4=2AsH_3+6ZnSO_4+3H_2O.$$

Now if this arseniëtted hydrogen is heated strongly, either by burning in the air, or by passing through a red-hot tube, it is decomposed, and metallic arsenic is deposited in the solid state.

258. Several forms of apparatus have been contrived for making use of this property in the detection of arsenic;—of these the following is in practice the simplest:—The bottle *a* (fig. 65) should be capable of containing six or eight ounces of water, and is connected by means of a perforated cork with the tubes *b* and *c*, the latter should be made of hard German glass, bent at a right angle, having the end *e* drawn off so as to diminish the aperture. A few fragments of zinc are placed in the bottle and covered with water; and when the cork with its tubes is attached, pour a little dilute sulphuric acid down the tube *b*, which should reach nearly to the bottom of the bottle, and allow the gas (hydrogen) to be given off for five minutes.† Then kindle the hydrogen at the mouth of the tube, and depress a porcelain plate (the lid of a Berlin crucible) upon the flame for a second or two; observe whether any brown stain is deposited upon it. This precaution is necessary, since some kinds of sulphuric acid, and also of zinc, contain traces of arsenic.‡ If no stain is produced, it may be assumed that the materials are pure.

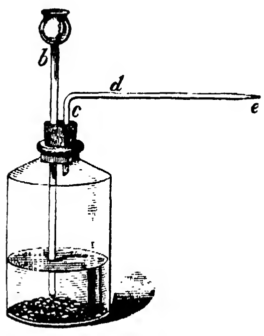

Fig. 65. Marsh's Test.

* It must be borne in mind that this gas, like most of the other compounds of arsenic, is highly poisonous; so that the experiment should never be performed in a close room, but in the open air or in a well-ventilated apartment.

† The reason why it is not safe to apply the light at once, is, that a mixture of hydrogen and common air is highly explosive, so that it is necessary to allow time for the whole of the common air to be expelled by the hydrogen; as otherwise serious injury might be caused by an explosion of the mixed gases.

‡ Antimony is also occasionally found in zinc.

259. The solution containing (or suspected to contain) arsenic, is now introduced through the tube *b*. If arsenic is present in the liquid, it will cause the formation of arsenictted hydrogen, which will impart a livid hue to the flame of the hydrogen; on depressing the porcelain plate again upon the flame, the metallic arsenic will be deposited in the form of a dark shining spot. By applying heat to the dark spots they are readily volatilized, and the fumes will be found to have the characteristic odour of garlic.

A few of these spots should be retained for further examination, and for comparison with those of antimony.

260. A deposit of metallic arsenic may also be obtained by strongly heating the tube through which the gas is passing, at the point *d* (fig. 65), when a lustrous mirror will be formed, not exactly at the heated point, but a quarter or half an inch beyond (in consequence of the volatility of arsenic). The portion of the tube which contains the deposit may be filed off and examined as directed at (246).*

261. Marsh's test, as just described, is so extremely delicate, that it is capable of detecting arsenic in a solution containing the millionth of its weight of the acid, and may be considered the most conclusive test which we possess. It is, however, liable to this objection, which is in practice easily overcome. It is found that antimony, when present in a mixture of zinc and dilute sulphuric acid, combines with the liberated hydrogen, precisely in the same way as arsenic, forming an analogous compound, called antimonietted hydrogen; which, when heated, is decomposed, and the metallic antimony is at the same time deposited. Hence it is extremely important that we should be able to distinguish accurately between them, as otherwise we should not be sure whether the crusts produced by Marsh's test were due to arsenic or antimony. One or two experiments are generally sufficient to enable us to do this.

If the H be evolved in an *alkaline* solution, as by sodium-amalgam in water, or by boiling Zn with KHO, no SbH_3 is evolved, but only AsH_3, which is easily recognised by the black stain produced on filter-paper moistened with $AgNO_3$.

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* For details respecting the application of Marsh's test in minute testing for toxicological purposes, the student is referred to *Bowman's Medical Chemistry*, or to *Guy's Forensic Medicine*.

262. For the purpose of comparison, empty the zinc and sulphuric acid from the bottle used for the arsenic experiments (258), wash it well, and substitute fresh zinc and acid. When the gas has been coming off about five minutes (note to 258), pour in a few drops of a solution of the double tartrate of antimony and potash (tartar-emetic), and apply heat as before at the point *d*. A crust of antimony will be deposited *at the heated point*, and not, as in the case of arsenic, at a little distance from it: this is owing to the antimony being less volatile than arsenic, and it will be found impossible to volatilize it by the heat of a common spirit lamp. In this manner, therefore, we are enabled in some measure to judge whether the stain is due to antimony or arsenic.

263. Light the jet of gas which issues from the aperture *e* as in (258), and hold over the flame a porcelain plate as before: a deposit of metallic antimony will be formed similar to that of arsenic, but blacker and less shining.

Prepare a few of these spots for comparison with those of arsenic formed in (259).

264. Apply the heat of a spirit-lamp to one of each kind of spot, and observe the superior volatility of the arsenic, and the garlic odour of its vapour.

265. (C) Moisten one of each kind of spot with yellow ammonium sulphide, on the end of a glass rod, and observe that the antimony *is immediately dissolved*, while the arsenic remains nearly unaffected for a considerable length of time. This is a most valuable means of distinguishing between them, and was first observed by Dr. Guy.

266. (C) If the spots be moistened with a solution of chloride of lime (*calcium hypochlorite*), the arsenic will dissolve, while the antimony will remain unaffected.

Reinsch's Test.

267. (C) This test is founded on the circumstance that when a metal, such as copper, is heated in a solution of another metal more electro-negative than itself, the latter is separated in the metallic state, and deposited on the surface of the former (as Cu_5As_2), which is at the same time dissolved in equivalent proportion. A little of the solution containing arsenic is acidified with a few drops of hydrochloric acid, and boiled in a test-tube with three or

four strips of clean copper foil: the arsenic, being more electro-negative than the copper, is deposited on the surface of the foil, and the whole is in this way separated from the solution.

$$As_2O_3+Cu_3+6HCl=As_2+3CuCl_2+3H_2O.$$

268. The appearance of a metallic deposit on the copper is not, however, necessarily a proof of the presence of arsenic, since other metals (as bismuth, silver, mercury, or antimony) would produce a similar incrustation, being all more electro-negative than copper. Arsenic, however, is readily distinguished from any of those in the following manner:—

269. Take the copper strips out of the solution, wash them with water, and dry them cautiously between folds of filtering paper; place them in a small tube sealed at one end (*not a test-tube*), and apply heat, when the arsenic will be volatilized, and, becoming oxidized by the air contained in the tube, will form a crystalline sublimate in the upper part (244).

270. Had the deposit on the copper consisted of silver, it would not have been volatilized when heated: if it were mercury, minute globules of the metal would have condensed in the cool part of the tube; and had it been bismuth or antimony, a higher degree of heat would have been necessary to sublime it; the sublimate would have been white and amorphous instead of crystalline;—and when treated with water, would prove insoluble, while the arsenious acid would dissolve, and the solution, on being tested, would show the presence of arsenic.*

* If the arsenic be present in the state of arsenic acid, the copper separates it with greater difficulty, and it is necessary to add a very large proportion of hydrochloric acid. A great obstacle to the use of this simple and delicate test is the difficulty of obtaining hydrochloric acid, and especially copper, perfectly free from arsenic. In testing these, a quantity of hydrochloric acid equal to that employed in testing the suspected substance, should be diluted with four times its bulk of water, boiled in a small flask, and a small strip of the copper introduced; if after boiling for 15 or 20 minutes, there is no deposit upon the copper, the hydrochloric acid may be considered sufficiently pure. Several of the strips of copper should be strongly heated in a small closed tube to prove that they will not afford crystals of arsenious acid.

No oxidizing agent (such as nitric or chloric acid) must be present in the liquid to be tested, or the copper will be dissolved. See *Bowman's Medical Chemistry*.

B. As Arsenic Acid (H_3AsO_4).

271. Mix a little arsenious acid with nitre, and heat it in a small tube sealed at the end. The nitre gives up a portion of its oxygen to the arsenic, forming potassium arseniate.

272. Dissolve the fused mass in water, and test it with *nitrate of silver:* a reddish-brown precipitate of arseniate of silver (Ag_3AsO_4) is thrown down, which is soluble in nitric acid, and also in ammonia. Compare (255).

273. Arsenic acid, even when mixed with hydrochloric acid, is not precipitated by hydrosulphuric acid unless the solution is boiled, or allowed to stand for some time. Compare (248).

274. For the detection of arsenic in organic mixtures, the student is referred to *Bowman's Medical Chemistry.*

CHAPTER III.

METALS BELONGING TO CLASS II.

Aluminium, Chromium, Zinc, Manganese, Iron, Nickel, and Cobalt.

275. The metals of the second class are distinguished from those of the first class in not being precipitated when their solutions, containing a slight excess of acid, are treated with hydrosulphuric acid.

SECTION I.

Aluminium (Al).

A solution of aluminium sulphate $Al_2(SO_4)_3$ or of alum $KAl(SO_4)_2$ may be used.

276. Salts of aluminium, when mixed with excess of

ammonium acetate, and boiled, yield a gelatinous precipitate of basic aluminium acetate.

277. (C) *Ammonium sulphide* gives a very transparent, gelatinous precipitate of aluminium hydrate, and hydrosulphuric acid is at the same time liberated.

$$Al_2(SO_4)_3+3(NH_4)_2S+6H_2O=Al_2H_6O_6+3(NH_4)_2SO_4+3H_2S.$$

278. (C) *Ammonia* throws down a similar gelatinous precipitate, consisting chiefly of aluminium hydrate, with a small admixture of a basic salt of aluminium; which is almost insoluble in an excess of ammonia. Compare (292).

279. *Potash* also gives a precipitate of aluminium hydrate, which, like that caused by ammonia, usually contains a little basic salt: it differs from it, however, in being entirely soluble in an excess of the precipitant. If the solution in potash be mixed with chloride of ammonium, the alumina is again precipitated, chloride of potassium being formed, and ammonia liberated.

280. *Phosphate of soda* produces a gelatinous precipitate of aluminium phosphate ($AlPO_4$); soluble in hydrochloric acid, and reprecipitated by ammonia; readily soluble in potash, and reprecipitated by acetic acid.

SECTION II.

Chromium (Cr).

A. *As Oxide of Chromium (sesquioxide of chromium or chromic oxide)* Cr_2O_3 *or some corresponding compound.*

A solution of chromium sulphate $Cr_2(SO_4)_3$ (or of chrome alum) may be used.

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281. (C) *Ammonium sulphide* throws down a greenish-blue precipitate of chromium hydrate ($Cr_2H_6O_6$).

282. (C) *Ammonia* also produces the same precipitate.*

283. (C) *Potash* also throws down the hydrate, which is soluble in a very slight excess, forming a green solu-

* A small portion of this precipitate redissolves in an excess of ammonia, forming a pale pinkish solution, but is again precipitated when the mixture is boiled.

tion: if this solution be boiled for a minute or two, the oxide is precipitated. If too large an excess of potash be employed, the boiling must be continued for some time before the precipitate appears.

284. (C) Oxide of chromium, when fused (on platinum foil) with nitrate of potash and a little carbonate of soda (the blowpipe flame being directed upon the under surface of the foil), yields a yellow mass. Here a portion of the oxygen of the nitrate combines with the oxide of chromium, converting it into chromic acid, which combines with the potash or soda, forming an alkaline chromate. If the mass be dissolved in water and acidulated with a little acetic acid, the solution will give with acetate of lead a bright yellow precipitate of chromate of lead.†

Chromic oxide, boiled with conc. HNO_3 and $KClO_3$, yields chromic acid, which may be detected by adding NH_3 in excess, acetic acid in excess, and lead acetate.

[For another blowpipe test, *see* (125).]

B. *As Chromic Acid* (CrO_3).

A solution of bichromate of potash ($K_2O.2CrO_3$) may be used.

285. (C) *Hydrochloric* and *hydrosulphuric acid* added in succession, the latter in large quantity, change the red colour of the solution to green (chromic chloride Cr_2Cl_6), the solution becoming turbid from separation of sulphur, the hydrogen of the hydrosulphuric acid having been oxidized. $2CrO_3+6HCl+3H_2S=Cr_2Cl_6+6H_2O+S_3$.

286. *Ammonium sulphide* gives, on boiling, a greenish-grey precipitate, consisting of a mixture of chromic hydrate and sulphur.

287. (C) *Chloride of barium* throws down a yellow precipitate of chromate of barium, $BaCrO_4$, soluble in dilute hydrochloric acid, but insoluble in acetic acid.

288. (C) *Nitrate of silver* produces a purple-red precipitate of chromate of silver (Ag_2CrO_4) soluble, though with some difficulty, in nitric acid.

* It is advisable to boil the acetic solution before testing with acetate of lead, in order to decompose any nitrite of potash.

289. (C) *Acetate of lead* gives a yellow precipitate of chrome yellow (chromate of lead $PbCrO_4$), insoluble in acetic acid.

SECTION III.

Zinc (Zn).

A solution of sulphate of zinc ($ZnSO_4$) may be used.

290. *Hydrosulphuric acid*, when added to a solution of a neutral salt of zinc, causes the precipitation of a portion of it as sulphide; but if the solution be mixed with hydrochloric acid, hydrosulphuric acid will produce no precipitate. $ZnSO_4 + H_2S = H_2SO_4 + ZnS$.

291. *Ammonium sulphide* gives a precipitate of sulphide of zinc, which, if the zinc salt be pure, is white;* but if, as is frequently the case, any iron is present, the precipitate will be more or less coloured, owing to the admixture of a little of the black sulphide of iron.

292. (C) *Ammonia* throws down a white gelatinous precipitate of zinc hydrate (ZnH_2O_2), which is very readily soluble in excess. The presence of ammoniacal salts (chloride of ammonium, for example,) prevents the precipitation. Compare (278).

If the ammoniacal solution be treated with hydrosulphuric acid, the white sulphide is thrown down.

Ferrocyanide of potassium added to the ammoniacal solution, gives a white precipitate of zinc ferrocyanide. (Zn_2Cy_6Fe).

293. (C) *Potash* behaves in the same manner as ammonia, giving a precipitate of zinc hydrate, soluble in excess. Hydrosulphuric acid throws down the white sulphide from the potash solution.

294. *Identification of metallic zinc.*—Pour a little dilute

* If the yellow ammonium sulphide be employed, of course any excess of this will impart a yellow appearance to the white precipitate suspended in the liquid. To identify a very small quantity of sulphide of zinc, collect it upon a filter, wash it, dissolve it in a very little hot nitric acid, add a trace of nitrate of cobalt (not enough to give a pink colour), then carbonate of soda in excess, boil for a minute or two, collect the precipitate of mixed carbonates of zinc and cobalt upon a filter, wash, and incinerate the filter on platinum foil. If zinc be present, the residue will be bright green after being strongly heated, especially when crushed with a glass rod.

nitric acid upon a very small piece of metallic zinc in a test-tube; it dissolves with effervescence, even in the cold. Prove the presence of zinc in the solution by adding excess of *ammonia* and *hydrosulphuric acid* (292).

[For the blowpipe tests, *see* (163, 164).]

SECTION IV.

Manganese (Mn).

A solution of sulphate of manganese ($MnSO_4$) may be used.

295. *Hydrosulphuric acid*, when added to an acidified solution (formed by adding a few drops of hydrochloric acid to a little of the solution), gives no precipitate.

296 (C) *Ammonium sulphide* gives a flesh-coloured precipitate of sulphide of manganese (MnS).

297. (C) *Ammonia* throws down a white precipitate of manganous hydrate, which if shaken with air becomes brown, owing to the formation of the sesquioxide.

$$2MnH_2O_2+O=Mn_2O_3+2H_2O.$$

If chloride of ammonium is present in the solution, it prevents the precipitation of the hydrate; owing to the formation of a double chloride of ammonium and manganese, which is not decomposed by ammonia. If the ammoniacal solution be exposed to the air, the brown sesquioxide is gradually precipitated.

298. *Potash* behaves as ammonia in solutions of manganese.

299. (C) *Carbonate of ammonia* throws down a white precipitate of carbonate of manganese, which is less prone to blacken on exposure than the hydrated oxide.

[For the blowpipe tests, *see* (137, 138).]

* Sesquioxide of manganese, manganic acid, and permanganic acid, would not escape detection by ammonium sulphide, which would convert them, by reduction, into sulphide of manganese. Manganic acid would be known by the green colour of its solutions, which changes to the red of permanganic acid on adding a little nitric acid. The red solutions of permanganic acid are bleached instantaneously by hydrosulphuric acid. The solutions of sesquioxide of manganese have a rose colour. Binoxide of manganese does not combine with acids, but evolves chlorine when heated with hydrochloric acid.

SECTION V..

Iron (Fe).

A. *As Protoxide* (*Ferrous oxide*, FeO), *or some corresponding compound.*

A solution of protosulphate of iron (ferrous sulphate, $FeSO_4$) may be used.

300. On account of the strong tendency of ferrous oxide to absorb oxygen on exposure to the air, and become ferric oxide, especially in aqueous solutions of its salts, it is difficult to retain the ferrous salts in solution without some admixture of ferric salts; so that in testing them, the indications of some of the reagents are frequently more or less different from those caused by a pure ferrous salt.

301. *Hydrosulphuric acid* produces no precipitate in acidified solutions of ferrous salts; a slight precipitation of sulphide takes place, however, in neutral solutions of some of them, especially when the acid with which it is in combination is a feeble one.

302. (C) *Ammonium sulphide*, when added to neutral solutions of ferrous salts, throws down a black precipitate of ferrous sulphide (FeS).

303. (C) *Ammonia* gives a precipitate of ferrous hydrate, which is at first nearly white, but almost immediately becomes greenish. If this precipitate be exposed to the air, it absorbs oxygen, and is changed into ferric hydrate ($2Fe_2O_3.3H_2O$), which has a reddish-brown or rust colour. Chloride and some other salts of ammonium prevent the precipitation of ferrous oxide by ammonia, forming a solution of a double salt of ammonium and iron, from which ferric hydrate is gradually precipitated on exposure to the air.

304. *Nitric acid*, on boiling, converts ferrous salts into ferric salts, which give a rust-coloured precipitate with ammonia.

$$6FeSO_4 + 8HNO_3 = 2Fe_2(SO_4)_3 + Fe_2(NO_3)_6 + 2NO$$

305. *Potash* behaves as ammonia.

306. (C) *Ferrocyanide of potassium* (K_4FeCy_6) throws down in solutions of ferrous salts, a precipitate which is at first almost white, but rapidly changes to pale blue; the colour becomes darker on exposure to the air, owing to the absorption of oxygen, and formation of Prussian blue.*

307. (C) *Ferricyanide of potassium* (K_3FeCy_6) produces in solutions of ferrous salts a beautiful dark-blue precipitate, similar in appearance and in composition to Prussian blue.† Compare (313).

[For the blowpipe test, *see* (133).]

B. *As Peroxide of Iron* (*Ferric oxide*, Fe_2O_3), *or some corresponding compound.*

A solution of the perchloride of iron (ferric chloride, Fe_2Cl_6) may be used.

308. *Hydrosulphuric acid* causes in neutral or acidified solutions of ferric salts, a precipitation of sulphur, which gives the solution a milky appearance.

$$Fe_2Cl_6 + H_2S = 2FeCl_2 + 2HCl + S.$$

309. (C) *Ammonium sulphide* separates the whole of the iron from solutions of ferric salts, as black sulphide. If the solution of iron is very dilute, no precipitate appears at first, but the solution becomes green, and if allowed to stand a considerable time, the sulphide gradually separates.

310. (C) *Ammonia* throws down the ferric hydrate ($2Fe_2O_3.3H_2O$), in the form of a bulky reddish-brown precipitate, which is insoluble in an excess of ammonia, and is unaffected by the presence of ammoniacal salts. Compare (303).

311. (C) *Potash* produces the same precipitate, which is insoluble in excess.

312. (C) *Ferrocyanide of potassium* produces in solutions of ferric salts a beautiful precipitate of soluble Prussian blue.‡

* $FeSO_4 + K_4(FeCy_6) = K_2SO_4 + FeK_2(FeCy_6)$.
$3FeK_2(FeCy_6) + O = Fe_3(FeCy_6)_2 + K_4(FeCy_6) + K_2O$.

† $3FeSO_4 + 2K_3(FeCy_6)_2 = Fe_3(FeCy_6)_2 + 3K_2SO_4$.

‡ $Fe_2Cl_6 + 2K_4(FeCy_6) = K_2Fe_2(FeCy_6)_2 + 6KCl$.

This is an extremely delicate and characteristic test for the ferric salts, as the precipitate is produced even in very dilute solutions. In testing for iron with ferrocyanide of potassium, however, it must be borne in mind that when added to a solution containing much free acid, it is partially decomposed, and a little Prussian blue is formed, even when no iron is present.* As the presence of free alkalies also interferes with the formation of the blue precipitate, solutions to be tested with it should be either neutral or slightly acid.

313. *Ferricyanide of potassium* produces no precipitate with ferric salts; it gives, however, a deep red-brown colour to the solution.† Compare (307).

314. (C) *Ammonium acetate* (prepared by adding acetic acid to ammonia till the solution is slightly acid) gives with ferric salts a red solution of ferric acetate. If this be diluted with much water and boiled, the whole of the iron is precipitated as a basic acetate. In this way iron may easily be separated from cobalt, nickel, manganese, and zinc.

Since the presence of a large excess of acid hinders the precipitation, it is advisable to nearly neutralize very acid solutions with carbonate of ammonia before adding the acetate of ammonia.

315. *Identification of metallic iron.*—Pour some dilute nitric acid upon two or three iron filings; accelerate the solution by heat, and prove the presence of ferric nitrate by testing with ammonia (310).

SECTION VI.

Nickel (Ni).

A solution of the sulphate of nickel ($NiSO_4$) may be used.

316. *Hydrosulphuric acid* causes no precipitate in

* This applies especially to mineral acids; in testing such solutions it is advisable that the addition of the ferrocyanide be preceded by that of acetate of ammonium, so that the mineral acid may be neutralized, and acetic acid liberated in its stead.

† Ferricyanide of potassium, which contains a little ferrocyanide (as is often the case with that which has been long kept in solution in the laboratory) gives a green instead of a brown colour with ferric salts.

acidified solutions of nickel; but if the solution is neutral, especially if the acid of the salt be a feeble one, a partial precipitation of sulphide of nickel takes place.

317. *Ammonium sulphide* (NiS) throws down, from neutral solutions, a black precipitate of sulphide of nickel which is slightly soluble in excess,* giving a brownish tint to the solution. Sulphide of nickel is sparingly soluble in dilute hydrochloric acid, but dissolves immediately on addition of nitric acid.

318. (C) *Ammonia* causes a pale green precipitate of nickel hydrate, which redissolves very easily, with a blue colour, when the ammonia is added in excess, owing to the formation of an ammoniated salt of nickel. If potash be added to the ammoniacal solution, it precipitates the nickel hydrate. The presence of ammoniacal salts in the nickel solution prevents the precipitation by ammonia.

319. (C) *Potash* also throws down the green nickel hydrate, which is insoluble in an excess of potash.

320. *Carbonate of ammonia* produces a precipitate of carbonate of nickel, which redissolves in excess to a blue liquid.

321. *Cyanide of potassium* throws down a precipitate of cyanide of nickel ($NiCy_2$), which has a yellowish-green colour: it redissolves in an excess of the alkaline cyanide, forming a dull yellow solution of the double cyanide of nickel and potassium (K_2NiCy_4), from which the cyanide of nickel is again precipitated on the addition of dilute hydrochloric acid. If the precipitate be boiled with potash, it is converted into oxide of nickel, cyanide of potassium being formed.

$$K_2NiCy_4+2KHO=NiO+4KCy+H_2O.$$

322. *Ferricyanide of potassium* produces a greenish-yellow precipitate of ferricyanide of nickel. Compare (329).

[For the blowpipe test, *see* (142).]

* That is, in excess of the ordinary yellow sulphide; the colourless freshly prepared sulphide does not dissolve it.

SECTION VII.

Cobalt (Co).

A solution of nitrate of cobalt, $Co(NO_3)_2$, may be used.

323. *Hydrosulphuric acid* gives in acidified solutions no precipitate. If the solution is neutral, a slight precipitation of the black sulphide of cobalt takes place.

324. (C) *Ammonium sulphide* throws down from neutral solutions a black precipitate of the sulphide of cobalt (CoS), which is insoluble in hydrochloric acid, but dissolves in nitric acid.*

325. (C) *Potash* throws down a precipitate of a blue colour, consisting of a basic salt of cobalt, which becomes dirty red on boiling, owing to the formation of cobalt hydrate. The precipitate is insoluble in excess of potash. Compare (319).

326. (C) *Ammonia* behaves as potash, but the precipitate readily redissolves in an excess, forming double salts of cobalt and ammonium, which are soluble in water. If the red ammoniacal solution is exposed to the air, it gradually becomes darker, owing to the absorption of oxygen, and formation of peroxide of cobalt.

327. *Carbonate of ammonia* gives a precipitate of carbonate of cobalt ($CoCO_3$), which readily dissolves in excess of the precipitant, yielding a red solution.

328. (C) *Cyanide of potassium*, when added to a solution of cobalt, gives a pale brown precipitate of cyanide of cobalt ($CoCy_2$), which when heated with an excess of cyanide of potassium, readily redissolves, forming cobalticyanide of potassium (K_3CoCy_6). The addition of hydrochloric acid causes no precipitate in this solution, thus distinguishing cobalt from nickel.† Compare (321).

329. *Ferricyanide of potassium* gives a purple-brown precipitate, or in very dilute solutions, a brown colour.

* Sulphide of cobalt is not so easily dissolved by an excess of yellow sulphide of ammonium as sulphide of nickel is.

† In order to insure the complete conversion of the cobalt into cobalticyanide of potassium the solution should be boiled for a minute or two. $2CoCy_2 + 8KCy + O = 2K_3(CoCy_6) + K_2O$.

330. If iron be present in combination with cyanogen, forming a ferrocyanide or ferricyanide, it may escape detection by the ordinary tests, unless the cyanogen be decomposed by ignition or otherwise. The addition of protosulphate or perchloride of iron would at once indicate the presence of ferro- or ferricyanogen (306, 307, 312, 313).

[For the blowpipe test, *see* (127).]

Remarks on the Separation of the Metals of Class II.

331. It will be seen, in Table X., that the metals of this class are separated in two subdivisions, of which one is precipitated by NH_3 in the presence of NH_4Cl, and comprises Al, Cr, and Fe, whilst the second, containing Zn, Mn, Ni, and Co, is precipitated subsequently by $(NH_4)_2S$. Some complication results from the circumstance that when Fe is present in large proportion, the ferric hydrate precipitated by NH_3 may possibly carry down with it all the Mn, Ni, and Co. Moreover, when Cr is present in large proportion it materially alters the behaviour of Fe. On these accounts the tables for the separation of the members of this class, which have to provide for the contingency of all the metals being present at the same time, involve many operations.

CHAPTER IV.

METALS BELONGING TO CLASS III.

Barium, Strontium, Calcium, and Magnesium.

332. These metals are distinguished from the preceding by not being precipitated from their solutions by hydrosulphuric acid or hydrosulphate of ammonia.

SECTION I.

Barium (Ba).

333. (C) A solution of chloride of barium ($BaCl_2$) may be used. *Carbonate of ammonia* gives a white precipitate of barium carbonate.

$$BaCl_2+(NH_4)_2CO_3=2NH_4Cl+BaCO_3.$$

The precipitate will be easily dissolved on adding dilute hydrochloric acid. $BaCO_3+2HCl=BaCl_2+H_2O+CO_2$.

334. *Dilute sulphuric acid* produces in solutions containing barium, even though they be very dilute, a copious white precipitate of barium sulphate.

$$BaCl_2+H_2SO_4=BaSO_4+2HCl.$$

This precipitate is insoluble in acids.

335. (C) Solution of *sulphate of lime* ($CaSO_4$)* causes an immediate precipitate of barium sulphate. This is the most convenient mode of applying a very dilute solution of a sulphate, since sulphate of lime requires about 500 times its weight of water to dissolve it. Compare (339).

336. (C) *Hydrofluosilicic acid* ($2HF.SiF_4$) causes a white highly crystalline precipitate of silicofluoride of barium, insoluble in dilute acids.

$$BaCl_2+2HF.SiF_4=BaF_2.SiF_4+2HCl.$$

336*a*. Potassium chromate or dichromate will produce a yellow precipitate of barium chromate ($BaCrO_4$), insoluble in acetic acid. This is taken advantage of in distinguishing and separating barium from strontium and calcium.

[For the blowpipe test for barium, *see* (114).]

SECTION II.

Strontium (Sr).

A solution of strontium nitrate ($Sr(NO_3)_2$) may be used.

337. (C) *Carbonate of ammonia* acts as with solutions of barium (333), carbonate of strontium ($SrCO_3$) being produced.

338. *Sulphuric acid* produces a white precipitate of strontium sulphate immediately, if the solution is not very dilute, and after standing a short time if it is so.

339. (C) Solution of *sulphate of lime* gives no immediate precipitate in solutions of strontium, but if allowed to stand, strontium sulphate gradually separates. Heat

* Calcium sulphate and sulphate of lime are identical.

hastens the precipitation. Strontium may thus be distinguished from barium (335).

[For the blowpipe test, *see* (155).]

SECTION III.

Calcium (Ca).

A solution of calcium chloride ($CaCl_2$) may be used.

340. (C) *Carbonate of ammonia* produces a white precipitate of carbonate of lime ($CaCO_3$), which becomes far less voluminous on heating the solution; it dissolves very readily in hydrochloric acid.

341. *Sulphuric acid*, when added to *concentrated* solutions containing calcium, gives an immediate white precipitate of calcium sulphate ($CaSO_4$).

If the solution is not concentrated, the precipitate may not appear at once, but will gradually separate in the form of minute crystals; and if the solution is very dilute, no precipitation will take place, because the sulphate, being soluble in about 500 times its weight of water, remains dissolved if sufficient water is present.

342. (C) After having thrown down the calcium sulphate, pour the mixture on a filter, and test the filtered solution with *ammonia and oxalate of ammonia* (343); sufficient of the sulphate will have been retained in solution to give a very perceptible precipitate with the oxalate.

343. (C) *Oxalate of ammonia* $(NH_4)_2C_2O_4$, is an extremely delicate test for lime, though it does not serve to distinguish it from baryta and strontia, which are also precipitated by it. When added to a solution containing it even in a highly diluted state, a copious white precipitate of oxalate of lime (CaC_2O_4) is produced.

It is necessary that the solution should contain no excess of acid, as the oxalate of lime is soluble in acid solutions; acetic acid, however, does not dissolve it.

[For the blowpipe test, *see* (118).]

SECTION IV.

Magnesium (Mg).

A solution of magnesium sulphate ($MgSO_4$) may be used.

344. *Ammonia*, when added to a neutral solution of magnesium, separates a portion of it in the form of magnesium hydrate (MgH_2O_2), which appears as a bulky white precipitate.

$$2MgSO_4+2NH_3+2H_2O=MgH_2O_2+(NH_4)_2Mg(SO_4)_2.$$

The rest of the magnesium remains in solution as a soluble double salt of ammonium and magnesium.

345. When *chloride of ammonium* has been added to a solution containing magnesium, *ammonia* no longer produces any precipitate, since the magnesium hydrate is soluble in chloride of ammonium.

346. (C) *Carbonate of ammonia* gives no precipitate unless the solution is boiled. The previous addition of chloride of ammonium entirely prevents the precipitation.

347. *Phosphate of soda* (Na_2HPO_4) gives a white precipitate of phosphate of magnesia ($MgHPO_4$), provided the solution is not very dilute, and especially on boiling.

348. (C) The addition of *chloride of ammonium* and *ammonia* to the magnesian solution, renders the phosphate of soda a far more delicate test than when used alone, because under those circumstances the double phosphate of ammonia and magnesia is produced, which is less soluble than the phosphate of magnesia, and is consequently thrown down from a more dilute solution than would furnish a precipitate with phosphate of soda alone. If the solution is very dilute, the precipitate does not appear at once, but if allowed to stand some little time, a crystalline deposit of the double phosphate gradually separates. Agitation of the liquid with a glass rod hastens the formation of this precipitate; and it is remarkable that if the tube be rubbed at all with the rod during agitation, lines of minute crystals are there first deposited.

$$MgSO_4+Na_2HPO_4+NH_3=Na_2SO_4+Mg(NH_4)PO_4.$$

As the double phosphate is readily soluble in an excess of acid, and slightly so in water, it is necessary that the solution should be pretty strongly ammoniacal.

It will be observed that in this test, the effect of ammoniacal salts in the solution is the reverse of that before described (345). When mixed with ammoniacal salts, indeed, magnesium can be precipitated only by a soluble phosphate.

Salts of barium, strontium, and calcium being also precipitated by phosphate of soda, this test cannot be relied on, unless the absence of those metals has been ascertained.

349. *Baryta water* (BaH_2O_2) (or *lime water*) throws down a white precipitate of magnesium hydrate. If the sulphate of magnesia be used, the insoluble sulphate of baryta will be thrown down at the same time.

CHAPTER V.

METALS BELONGING TO CLASS IV.

Potassium, Sodium, and Ammonium.

350. The three metals belonging to this class are chiefly characterized by the solubility in water of most of their compounds, and the consequent difficulty of obtaining them in an insoluble form, and of separating them from one another in the shape of precipitates.

351. They are distinguished from all other metals by producing no precipitate when tested with either of the three classifying tests—viz., hydrosulphuric acid, hydrosulphate of ammonia, and carbonate of ammonia, their sulphides and carbonates being all soluble in water.

352. Solutions of the oxides of these metals (alkalies), and of their carbonates, are alkaline to test paper, turning reddened litmus blue, and turmeric brown.

SECTION I.

Potassium (K).

353. Evaporate a drop of the solution of a compound of potassium (chloride of potassium or nitrate of potash, for

example) upon a slip of thin glass or of platinum foil, and observe that the dry residue is *fixed*—that is, will not volatilize upon the continued application of heat.

354. (C) Place a drop of the solution (*e.g.*, potassium nitrate), with a glass rod, upon a piece of glass, add a drop of *hydrochloric acid*, and another of *platinic chloride* ($PtCl_4$). Stir them well together with the rod, and observe the formation of a yellow crystalline precipitate of the double chloride of platinum and potassium ($2KCl.PtCl_4$) especially upon the lines where the glass has been rubbed with the rod.

If the solution of potassium be very dilute, a considerable time may be required for the production of this precipitate.

355. Repeat this experiment with iodide of potassium; notice the red colour due to platinic iodide, and the delay in the appearance of the precipitate.

356. (C) Test a drop of the solution of potassium nitrate upon a piece of glass, with a strong solution of *tartaric acid* ($H_2C_4H_4O_6$). On stirring briskly, a very crystalline white precipitate of bitartrate of potash will be formed, especially upon the lines of friction.

$$KNO_3+H_2C_4H_4O_6=HNO_3+KHC_4H_4O_6.$$

A drop of hydrochloric acid will redissolve it, so that this test can only be depended on when the original solution is neutral or alkaline. Before testing an acid solution with tartaric acid, it should be neutralized with carbonate of soda, but the tartaric acid must always be added in excess.

[The blowpipe test will be found at (149).]

SECTION II.

Sodium (Na).

357. Evaporate a drop of a solution containing sodium (*e.g.*, sulphate of soda) upon glass or platinum; the dry residue will not volatilize when heated.

358. (C) Test a drop of the solution on a piece of glass with *antimoniate of potash*.* On stirring briskly, a white

* The bimetantimoniate of potash of Frémy, ($K_2H_2Sb_2O_7$), which produces, in a solution containing sodium, a precipitate of sodium bimetantimoniate ($Na_2H_2Sb_2O_7$).

crystalline precipitate of antimoniate of soda will be deposited, especially upon the lines of friction.

Test a drop of any dilute acid (as hydrochloric) with antimoniate of potash, and observe that it gives an amorphous precipitate of hydrated antimonic acid, showing that an acid solution must not be tested for soda until it has been neutralized with potash or carbonate of potash.

[The blowpipe test for sodium will be found at (154).]

SECTION III.

Ammonium (NH_4).*

359. (C) Evaporate a drop of solution of chloride of ammonium upon a piece of glass or of platinum foil, and continue to heat the dry residue; it will entirely volatilize. (Compare 353, 357.)

360. (C) Boil some solution of chloride of ammonium in a test-tube; observe that no smell of ammonia is perceived, but add a little *potash*, and the liberated ammonia will at once be recognised by its odour and by its restoring the blue colour to red litmus paper, which should only be exposed to the vapour, and not allowed to touch the tube.

$$NH_4Cl + KHO = NH_3 + KCl + H_2O.$$

361. Test a drop of solution of chloride of ammonium upon a slip of glass with *platinic chloride*, which will produce a precipitate of the double chloride of platinum and ammonium ($2NH_4Cl.PtCl_4$) very similar to that obtained with a compound of potassium (354).

362. Mix a little solution of carbonate of ammonia† with *tartaric acid* upon a piece of glass; observe how easily the precipitate of bitartrate of ammonia might be mistaken for bitartrate of potash (356).

363. (*Nessler's test*). To a solution of mercuric chloride, add iodide of potassium until the red precipitate first formed is just dissolved; then add excess of potash. The solution thus prepared, when added to a liquid containing even a very minute quantity of ammonia or of one of its salts, will give a rich brown precipitate composed of mercury, iodine, and nitrogen. ($NHg_2I.H_2O$?).

* It will be remembered that this metal is not known in the uncombined state.

† If chloride of ammonium were employed it would be difficult to obtain a precipitate, since it is very readily soluble in the hydrochloric acid liberated by the decomposition.

CHAPTER VI.

NON-METALLIC BODIES.

SECTION I.

Nitrogen.

364. The most frequent forms of combination in which this element is met with are ammonia and its salts, nitric acid and the nitrates, cyanogen (CN) and its compounds, and nitrogenized organic substances which contain, beside nitrogen, carbon, hydrogen, and sometimes oxygen and sulphur. A method of recognising nitrogen in this last form has been given at (145), and the tests for ammonia have been already described in the section on ammonium, the recognition of cyanogen will be treated of hereafter. It remains to be seen how nitrogen is recognised when in the form of nitric acid or a nitrate.

Nitric Acid.

365. Heat a little powdered nitrate of potash (KNO_3) or some other nitrate, with *concentrated sulphuric acid;* vapours of nitric acid are evolved, mingled usually with red-brown vapours of nitric peroxide (84).

$$2HNO_3 = H_2O + 2NO_2 + O.$$

366. (C) Mix some solution of nitrate of potash with about an equal volume of *concentrated sulphuric acid*, add a few *copper filings*, and apply heat; red-brown vapours are produced, which are seen to the greatest advantage by looking down the tube. In this experiment, the nitric acid, which is set free by the sulphuric acid, is deoxidized by the copper, being converted into nitric oxide, which absorbs oxygen when it issues into the air, forming red fumes of nitric peroxide (22).

367. (C) Shake a small crystal of *protosulphate of iron* with solution of nitrate of potash for a few seconds, without waiting till it is entirely dissolved, and pour *con-*

centrated sulphuric acid very carefully down the side of the tube (slightly inclined), so that it may form a distinct layer at the bottom; a brown or pink band will appear where the two layers of liquid are in contact. If the quantity of nitric acid present be very minute, several minutes may elapse before the reaction is perceptible. The brown compound ($4FeSO_4.NO$) is formed by the union of ferrous sulphate with nitric oxide, the rest of the oxygen of the nitric acid having combined with another portion of the ferrous sulphate to convert it into ferric sulphate.*

If the tube be shaken or heated, the brown compound is decomposed, and the colour disappears.

[For the blowpipe tests *see* (143, 144).]

SECTION II.

Sulphur.

368. Sulphur is most frequently met with in the form of sulphuric acid or a sulphate; much less frequently as sulphurous acid or a sulphite, or as a hyposulphite (a salt of hyposulphurous acid). It also commonly occurs as hydrosulphuric acid or sulphuretted hydrogen, and as a sulphide (a combination of sulphur with a metal).

Sulphuric Acid.

369. Mix a few drops of strong sulphuric acid (oil of vitriol) with about an equal quantity of water, in a test-tube, and observe the heat evolved. Dilute the mixture with three or four volumes of water, place a drop upon a piece of filter paper, and dry it without scorching the paper; observe the carbonization of that part of the paper which was moistened with the acid.

370. (C) Test a solution of sulphate of soda, or some other sulphate, with *chloride of barium;* a heavy white precipitate of sulphate of baryta is obtained, which is insoluble in dilute hydrochloric acid.

$$Na_2SO_4+BaCl_2=BaSO_4+2NaCl.$$

[For the blowpipe test, *see* (158).]

* $2KNO_3 + 6FeSO_4 + 4H_2SO_4 = K_2SO_4 + 3Fe_2(SO_4)_3 + 4H_2O + 2NO.$

Sulphurous Acid.

371. (C) Heat a little sulphite of soda with *dilute hydrochloric acid;* sulphurous acid will be disengaged, and may be recognised by its odour of burning sulphur.

$$Na_2SO_3+2HCl=H_2O+SO_2+2NaCl.$$

372. (C) Acidulate a solution of sulphite of soda strongly with *hydrochloric acid,* and introduce a fragment of *zinc;* sulphuretted hydrogen will be evolved, and may be known by its odour, and by its action upon paper moistened with acetate of lead (378).

$$Na_2SO_3+8HCl+Zn_3=H_2S+2NaCl+3ZnCl_2+3H_2O.$$

373. *Chloride of barium* and *chloride of calcium* produce in the solution of a sulphite (*e.g.*, sulphite of soda) white precipitates ($BaSO_3$ and $CaSO_3$) which are soluble in dilute hydrochloric acid.*

374. *Nitrate of silver* produces a white precipitate of sulphite of silver (Ag_2SO_3), which becomes reduced to the metallic state when heated in the liquid, sulphuric acid being produced. $Ag_2SO_3+H_2O=Ag_2+H_2SO_4$.

Hyposulphurous Acid.

375. (C) Add a little *hydrochloric acid* to solution of hyposulphite of soda; after a short time the solution becomes milky, from separation of sulphur, and evolves the odour of sulphurous acid. The change is accelerated by heat. $Na_2S_2O_3+2HCl=2NaCl+H_2O+SO_2+S$.

376. Prepare a little chloride of silver by precipitating nitrate of silver with chloride of sodium, wash it by decantation, and shake it with solution of hyposulphite of soda, which will readily dissolve it, producing a very sweet solution. $AgCl+Na_2S_2O_3=NaCl+NaAgS_2O_3$.

376*a*. (C) Nitrate of silver produces, with a solution of hyposulphite of soda, a precipitate which is at first white (hyposulphite of silver), but rapidly passes through yellow and brown, finally becoming black (sulphide of silver).

$$Ag_2S_2O_3+H_2O=Ag_2S+H_2SO_4.$$

* Since commercial sulphites nearly always contain sulphates, the precipitate will seldom be found to dissolve entirely.

Hydrosulphuric Acid and Metallic Sulphides.

377. (C) *Acetate of lead* gives, with solution of hydrosulphuric acid or a sulphide, a black precipitate of sulphide of lead.

$$Pb(C_2H_3O_2)_2+H_2S=PbS+2HC_2H_3O_2.$$

378. (C). Pour a little *dilute hydrochloric acid* upon a small fragment of sulphide of iron in a test-tube; hydrosulphuric acid is evolved, which may be known by its odour, and if a piece of filter paper moistened with solution of acetate of lead be held at the mouth of the tube, it will be coated with a black shining deposit of sulphide of lead. $FeS+2HCl=H_2S+FeCl_2$.

379. Add a little *dilute hydrochloric acid* to a drop or two of yellow ammonium sulphide, hydrosulphuric acid is given off, and the liquid becomes milky from the separation of sulphur.

When ammonium sulphide is first prepared, it is colourless, but is gradually decomposed by the atmospheric oxygen, which combines with its hydrogen to form water, leaving ammonium disulphide, which has a yellow colour, and is precipitated on adding an acid.*

380. Boil a little sulphide of iron with *concentrated nitric acid*; the metal is oxidized and dissolved, whilst the sulphur separates in grey flakes, which rise to the surface of the liquid, and eventually fuse into yellow globules. Dilute the solution with water, filter it, and test with chloride of barium for the sulphuric acid which has been formed by the oxidation of a portion of the sulphur.†

[For the blowpipe test, *see* (157).]

* $2(NH_4)_2S + O = (NH_4)_2S_2 + 2NH_3 + H_2O.$
$(NH_4)_2S_2 + 2HCl = 2NH_4Cl + H_2S + S.$

† Very minute quantities of the alkaline sulphides may be detected by *nitroprusside of sodium*, which produces a fine purple colouration. Sulphur in organic substances (*e.g.*, in hair) may be detected by this test if the substance be fused with carbonate of soda on platinum wire in the reducing flame of the blowpipe, and the sulphide of sodium thus produced be dissolved in water. Sulphates might be detected in the same way by fusing them with carbonate of soda and charcoal.

SECTION III.

Phosphorus.

381. This element is generally met with in the form of tribasic phosphoric acid or a tribasic phosphate.

Tribasic Phosphoric Acid (*Orthophosphoric*, H_3PO_4).

A solution of common phosphate of soda (Na_2HPO_4) may be used.

382. (C) Mix a little solution of *sulphate of magnesia* with *chloride of ammonium* and *ammonia* (345), and add the mixture to a solution of phosphate of soda; a white crystalline precipitate of phosphate of magnesia and ammonia (348) will be produced. If the solution be dilute, the precipitate is formed only after standing; its deposition is promoted by rubbing the side of the tube with a glass rod. (Arsenic acid gives a similar reaction.)*

383. (C) *Perchloride of iron* gives a nearly white precipitate of phosphate of iron (ferric phosphate) which is insoluble in acetic acid, but dissolves readily in hydrochloric acid.

$$2Na_2HPO_4 + Fe_2Cl_6 = 2FePO_4 + 4NaCl + 2HCl.$$

(Arsenic acid gives a similar reaction, but ferric arseniate, after washing, dissolves in ammonia, while ferric phosphate is decomposed and becomes brown.)

384. (C) Dissolve a very little bone-ash (phosphate of lime, $Ca_3(PO_4)_2$) in as little dilute hydrochloric acid as possible, add a little perchloride of iron, and some acetate of potash or acetate of ammonia (prepared by mixing ammonia with acetic acid to acid reaction); collect the precipitated phosphate of iron upon a filter, wash it, dissolve off the filter with a little warm dilute hydrochloric acid, mix the solution with excess of ammonia and hydro-

* But the arseniate of magnesia and ammonia, when washed and treated with nitrate of silver, becomes red-brown, whilst the phosphate becomes yellow.

sulphate of ammonia, boil, filter off the black sulphide of iron, and test the solution for phosphoric acid as in (382).

385. *Chloride of barium* produces in a solution of phosphate of soda a white precipitate of phosphate of baryta ($BaHPO_4$) which is soluble in dilute hydrochloric acid. Compare (370).

386. *Chloride of calcium* gives a white precipitate of phosphate of lime ($CaHPO_4$), which readily dissolves in acetic acid (424); the solution gives a white precipitate with perchloride of iron, which must be added in very small quantity.

387. (C) *Nitrate of silver* gives a yellow precipitate of tribasic phosphate of silver, which is soluble both in ammonia and nitric acid. (Arsenic acid gives a brown precipitate.)

388. (C) Dissolve a very little bone-ash (phosphate of lime) in dilute nitric acid, and add excess of *molybdate of ammonia* (NH_4HMoO_4). The solution becomes yellow, and when heated deposits a yellow precipitate containing phosphoric acid, molybdic acid and ammonia.* (Arsenic acid gives a somewhat similar reaction, but only on boiling the liquid.)

389. Heat a crystal of common phosphate of soda to redness on platinum foil or wire; dissolve it in water, and test with nitrate of silver. Instead of the yellow orthophosphate, it will give a white granular precipitate of the pyrophosphate ($Ag_4P_2O_7$) corresponding to the pyrophosphate of sodium ($Na_4P_2O_7$) into which the common phosphate has been converted by the action of heat.

$$2Na_2HPO_4 = Na_4P_2O_7 + H_2O.$$

[For the blowpipe test, *see* (148).]

* This is by far the readiest test for phosphoric acid in combination with the earths or alkaline earths. It is necessary, however, that they should be dissolved in nitric acid, since hydrochloric acid hinders the formation of the yellow precipitate; and the temperature should not be raised above 100° F., lest arsenic acid be precipitated. The molybdate of ammonia must be added in large proportion relatively to the phosphoric acid present. A yellow colour only is not a proof of the presence of phosphoric acid. Silicic acid gives a yellow colour on heating.

SECTION IV.

Chlorine.

390. The most frequently occurring forms of combination of chlorine are hydrochloric acid, and the chlorides formed by the union of chlorine with a metal. It is also occasionally met with in combination with oxygen, as chloric acid and the chlorates, or hypochlorous acid and the hypochlorites.

Hydrochloric acid and metallic chlorides.

391. (C) Add *nitrate of silver* to a solution of chloride of sodium, or any other chloride; a white curdy precipitate of chloride of silver will separate, unless the solution be extremely dilute, when a milkiness only would be perceived.

Pour a part of the liquid containing the precipitate into another tube, and test the remainder with nitric acid, which will not dissolve it, although it may cause it to shrink together and undergo an apparent diminution.

Shake the other portion with ammonia, which will immediately dissolve it; on adding nitric acid to the ammoniacal solution, the chloride of silver is again precipitated.

392. (C) Pour a little *concentrated sulphuric acid* upon solid chloride of sodium; it froths and evolves hydrochloric acid, which may be known by its odour, and by the grey fumes which it produces in moist air* (56).

393 (C) Heat a little solid chloride of sodium, or other chloride to be tested, with *dilute sulphuric acid* and *binoxide of manganese;* chlorine is evolved, and may be recognised by its odour, and by its property of bleaching moistened test-papers.

$$2NaCl+MnO_2+2H_2SO_4=Na_2SO_4+MnSO_4+2H_2O+Cl_2.$$

Chloric Acid.

394. (C) Place a small fragment of chlorate of potash ($KClO_3$) in a test-tube, and pour upon it a few drops of

* A bromide would yield brown vapour of bromine, having an extremely irritating odour.

strong sulphuric acid; the chlorate is decomposed, yielding perchloric acid and chloric peroxide, which gives the mixture a red or yellow colour, and escapes in the form of a greenish-yellow gas of very peculiar odour.

$$3KClO_3+3H_2SO_4=3KHSO_4+HClO_4+2ClO_2+H_2O.$$

On applying a gentle heat to the mixture, sharp detonations are often perceived, arising from the decomposition of the peroxide of chlorine into its elements. If any chloride be present in the chlorate, the detonations often take place in the cold, in consequence of the mutual decomposition between the hydrochloric and chloric acids which are then evolved.

395. (C) Heat a little of the chlorate in a small sealed tube, *not a test-tube;* it is decomposed, and if the heat be continued long enough, the whole of the oxygen is given off, chloride of potassium being left (44). The disengagement of oxygen may be proved by introducing into the tube a match with a spark at the end.

Dissolve the residue out of the tube with water, and test the solution with nitrate of silver, which will give a precipitate of chloride of silver, insoluble in nitric acid.

395*a*. Add to the solution of chlorate of potash some nitrate of silver, to be sure that it contains no chloride. To another portion, add two or three drops of dilute sulphuric acid, and a piece of zinc; the hydrogen then produced will convert the chloric acid into hydrochloric acid, which may be detected, after a minute or two, with nitrate of silver (391). Heat hastens the conversion.

[For the blowpipe tests, *see* (121, 123).]

Hypochlorous Acid.

Solution of chloride of lime (hypochlorite of lime $Ca(ClO)_2$ with chloride of calcium and free lime) may be used.

396. (C). Solutions of the hypochlorites bleach indigo, litmus, and many other organic colouring matters very readily, when mixed with a little dilute sulphuric acid,

which liberates chlorine from the commercial hypochlorites, recognised by its odour.*

397. (C) Sulphate of manganese gives a black precipitate of binoxide of manganese.

$$Ca(ClO)_2 + 2MnSO_4 + 2CaH_2O_2 = 2CaSO_4 + CaCl_2 + 2MnO_2 + 2H_2O.$$

SECTION V.

Iodine.

398. We are most likely to meet with iodine, either in the free state, or in combination with hydrogen as hydriodic acid, or with a metal as an iodide.

Free iodine would be at once recognised by its resembling black lead in appearance, and furnishing violet vapours of peculiar odour when heated.

Hydriodic acid and metallic iodides.

A solution of iodide of potassium may be used.

399. (C) *Starch* added to a solution of an iodide, gives no reaction unless the iodine is set free by the addition of some oxidizing agent. This may be effected by *concentrated nitric acid*, which should be added drop by drop, when the purple or blue colour of the iodized starch will be perceived. Strictly speaking, the action is not due to the nitric acid, but to the nitrous acid, HNO_2 (?), which is contained in the ordinary specimens of the acid.

400. (C) *Concentrated sulphuric acid*, heated with an iodide, liberates iodine, recognised by its violet-coloured vapour, which imparts a blue colour to paper impregnated with starch.†

401. (C) *Nitrate of silver* gives a pale yellow precipitate

* The commercial hypochlorites always contain chlorides. A pure hypochlorite would disengage hypochlorous acid, the smell of which differs somewhat from that of chlorine.

$$Ca(ClO)_2 + CaCl_2 + 2H_2SO_4 = 2CaSO_4 + Cl_4 + 2H_2O.$$

† Some hydrosulphuric acid is evolved at the same time.

$$8KI + 5H_2SO_4 = 4K_2SO_4 + I_8 + H_2S + 4H_2O.$$

of iodide of silver (AgI) insoluble in nitric acid and in ammonia.

402. (C) *Perchloride of mercury* gives a brilliant red precipitate of iodide of mercury (HgI_2), soluble in an excess either of the perchloride or of iodide of potassium.

403. *Platinic chloride* produces a dark brownish-red colour.*

SECTION VI.

Fluorine.

404. This element is likely to be met with either as hydrofluoric acid, or as the fluoride of a metal.

Hydrofluoric acid and metallic fluorides.

405. (C) Moisten a little fluor spar (CaF_2) upon a piece of glass with strong sulphuric acid, heat gently, and wash off the mass after a few minutes, when the glass beneath will be found corroded by the hydrofluoric acid.

$$CaF_2+H_2SO_4=CaSO_4+2HF.$$

406. (C) Mix some fluor spar with *sand* (SiO_2), and heat the mixture with *concentrated sulphuric acid* in a dry test-tube furnished with a perforated cork, carrying a piece of glass tube moistened in the inside;† fluoride of silicon will be evolved, and will be decomposed by the water, depositing a white coating of silica (SiO_2) upon the sides of the moistened tube.

$$2CaF_2+SiO_2+2H_2SO_4=2CaSO_4+2H_2O+SiF_4;$$
$$3SiF_4+2H_2O=SiO_2+2(2HF.SiF_4).$$

407. Fuse upon platinum foil a mixture of very finely powdered fluor spar with three or four parts of carbonate of soda; boil the fused mass upon the foil in a dish with water, to dissolve out the fluoride of sodium (NaF), and thus separate it from the carbonate of lime. Filter the solution, and neutralize it carefully with acetic acid.

408. *Chloride of barium* added to a portion of this solution gives a translucent precipitate of fluoride of barium (BaF_2), soluble in dilute hydrochloric acid.

* Bichloride of palladium is often a useful test for iodides, with which it produces a black precipitate (PdI_2).

† Or place a broken test-tube, moistened inside, with its border in contact with that of the tube containing the fluoride, so that the gas evolved must pass up through it. Or hold a wet glass rod in the fumes.

409. *Chloride of calcium* gives a similar precipitate, almost insoluble in acetic acid.

SECTION VII.

Boron.

410. The ordinary forms of combination in which boron is met with are boracic acid and the borates.

Boracic Acid.

411. (C) Add *dilute hydrochloric acid*, drop by drop, to a solution of borax (biborate of soda, $Na_2O.2B_2O_3$), until it distinctly reddens blue litmus paper; dip one end of a slip of *turmeric paper* into the acid solution, and dry it at a little distance above the flame, being careful not to scorch it; the yellow colour of the turmeric will be changed to a bright orange-red, which will become blue-green with potash.

412. (C) Mix a solution of borax, or other borate to be tested, in a small dish, with *strong sulphuric acid* and alcohol, kindle the mixture, by means of a glass rod dipped into it, and stir it well, in a dark place; the flame will be tinged with green, especially at the edge, boracic ether being formed and burning with a green flame.*

413. Make a very strong solution of borax, by boiling with water, allow it to cool, and add *strong hydrochloric acid;* feathery crystals of boracic acid (H_3BO_3) will be deposited.

414. *Chloride of barium*, added to a solution of borax, precipitates biborate of baryta ($BaO.2B_2O_3$), which is readily soluble in dilute hydrochloric acid. Compare (376).

415. *Chloride of calcium* gives a white precipitate of biborate of lime, readily soluble in acetic acid. Compare (424).

Both this precipitate and the preceding one are soluble in chloride of ammonium, so that when dissolved in hydrochloric acid, they are not generally reprecipitated by ammonia.

* If copper be present it may also impart a green flame, and must be separated before applying this test.

416. *Nitrate of silver* gives a white precipitate of borate of silver which becomes brown when boiled, and is easily soluble in nitric acid. Compare (391).

[For the blowpipe test, *see* (117).]

SECTION VIII.

Silicon.

417. Silicic acid and the silicates are the forms in which this element is generally met with.

Silicic Acid.

418. (C) Mix a little finely powdered sand (SiO_2) (insoluble variety of silica) with four parts of dry *carbonate of soda*, and fuse the mixture for two or three minutes upon platinum foil, directing the blowpipe flame upon the under side; effervescence takes place, from the carbonic acid, and silicate of soda is formed.

Soak the platinum foil in water (in a dish), and the mass will dissolve; acidify the solution with hydrochloric acid, and divide it into two parts.

419. (C) Evaporate one part (acidulated with hydrochloric acid) to dryness, in a dish, and treat the residue with *diluted hydrochloric acid;* the chloride of sodium will be dissolved,* but white flakes of insoluble silica (SiO_2) will be left, and may be seen when the liquid is poured out into a test-tube.

420. To the other part add a slight excess of ammonia; soluble silica will separate as a gelatinous precipitate, which might easily be mistaken for alumina (278).

[For the blowpipe tests, *see* (150, 151).]

SECTION IX.

Carbon.

421. The forms of combination in which carbon is commonly met with in the analysis of inorganic substances

* Water would have sufficed to dissolve the chloride of sodium, but sand generally contains alumina and iron, which the hydrochloric acid will dissolve.

are carbonic acid and the carbonates; but since this element is present in all organic (*i.e.*, animal and vegetable) substances, the various forms of combination in which it may present itself are far too numerous to be considered in this work. There are, however, a few other acids containing carbon which are liable to be encountered in the ordinary course of analysis, and are here enumerated, their chief source, whether natural or artificial, being at the same time recalled to the mind of the student.

Oxalic acid; from wood sorrel; rhubarb; action of nitric acid on starch or sugar, and of hydrate of potash on sawdust.
Acetic acid; from vinegar; and distillation of wood.
Hydrocyanic acid; from bitter almonds; prussiate of potash.
Hydroferrocyanic acid; from yellow prussiate of potash.
Tartaric acid; from juice of the grape.
Citric acid; from lemon-juice.
Tannic acid; from gall nuts.
Gallic acid; from gall nuts.
Benzoic acid; from gum benzoin.

Carbonic Acid (CO_2 or H_2CO_3?).

422. (C) Add *dilute hydrochloric acid* to solution of carbonate of soda; notice the effervescence, and the faint odour of the carbonic acid evolved. Put a little powdered marble or chalk into a test-tube, pour upon it some dilute hydrochloric acid, and incline the mouth of the tube over that of another, half filled with lime-water, so as to pour the carbonic acid gas into the second tube, but not any of the liquid; close the tube containing lime-water (calcium hydrate) with the thumb, and shake it, when it will become milky, from the separation of carbonate of lime.

$$CaH_2O_2+CO_2=CaCO_3+H_2O.$$

If the quantity of carbonic acid be very small, it is better to pass it into lime-water, as represented in fig. 66; *a* representing

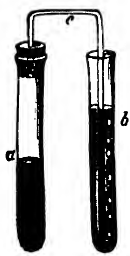


Fig. 66.

the test-tube containing the carbonate, from which the gas is conducted by the bent tube *c* into the lime-water in *b*; a very gentle heat may be applied to promote the disengagement of the carbonic acid.*

423. *Chloride of barium* or *Chloride of calcium* added to a solution of a carbonate, will produce a white precipitate of carbonate of baryta or of lime, readily soluble, with effervescence, in acetic acid.

$$BaCl_2+Na_2CO_3=2NaCl+BaCO_3.$$

Oxalic Acid ($H_2C_2O_4$).

424. (C) Add some *chloride of calcium* to a solution of an oxalate (*e.g.*, oxalate of ammonia); a white precipitate of oxalate of lime is produced, even though the solution be very dilute (343).

The precipitate will be found to be insoluble in acetic acid, but readily soluble in hydrochloric acid.

425. *Chloride of barium*, added to oxalate of ammonia, will give a white precipitate of oxalate of baryta, which is soluble in dilute hydrochloric acid.

$$BaCl_2+(NH_4)_2C_2O_4=2NH_4Cl+BaC_2O_4.$$

426. Heat a little crystallized oxalic acid in a small tube; observe that a great part volatilizes unchanged, condensing in fine needles on the cooler part of the tube.

427. (C.) Pour a little *strong sulphuric acid* upon some oxalic acid, or any oxalate to be tested, and apply heat; effervescence ensues, from the disengagement of carbonic acid and carbonic oxide (35).

The carbonic oxide may be recognised by its burning with a blue flame when the mouth of the tube is applied to a light.

428. Heat a little oxalate of lime with dilute hydrochloric acid, which will dissolve it without effervescence; neutralize the acid with ammonia, and the oxalate will be precipitated.

429. Place some oxalate of lime (CaC_2O_4) upon platinum foil, and heat it to low redness; the salt is scarcely changed in appearance, but if it be now moistened with

* Some natural carbonates, especially dolomite (carbonate of lime and magnesia), and native carbonate of iron, give scarcely any effervescence with hydrochloric acid in the cold.

hydrochloric acid, it will effervesce violently, having been converted into carbonate of lime by calcination.

$$CaC_2O_4 = CaCO_3 + CO.$$

If the solution in hydrochloric acid be boiled, to expel free carbonic acid, and neutralized with ammonia, no precipitate will occur if the oxalate has been completely decomposed.

430. If oxalate of lime be heated with *dilute sulphuric acid*, no effervescence takes place, but on adding a little *black oxide of manganese* there will be a brisk effervescence; due to the escape of carbonic acid gas produced by the oxidation of the oxalic acid at the expense of the oxide of manganese.

$$CaC_2O_4 + MnO_2 + 2H_2SO_4 = CaSO_4 + MnSO_4 + 2CO_2 + 2H_2O.$$

Acetic Acid ($HC_2H_3O_2$).

431. (C) Heat a little acetate of lead in a small tube, sealed at one end, and observe the peculiar odour of acetone (C_3H_6O) which it emits.

432. (C) Pour a little *strong sulphuric acid* upon some acetate of lead, or other acetate to be tested, in a test-tube, and apply heat, when acetic acid will be evolved, which may be recognised by its odour.

$$Pb(C_2H_3O_2)_2 + H_2SO_4 = PbSO_4 + 2(HC_2H_3O_2).$$

433. (C) Repeat the last experiment, with the addition of a little *alcohol;* the peculiar agreeable odour of acetic ether will be perceived, especially when the mixture has cooled. $HC_2H_3O_2 + C_2H_5HO = C_2H_5C_2H_3O_2 + H_2O$.

434. Add ammonia, drop by drop, to acetic acid, till the liquid is slightly alkaline, evaporate it gently in a dish till the alkaline reaction is no longer perceptible, and add *perchloride of iron;* a red colour (but no precipitate) is produced, due to the formation of ferric acetate;* the colour does not disappear on adding perchloride of mercury, which bleaches the red colour due to hydrosulphocyanic acid.

435. Add *nitrate of silver* to some acetic acid; no precipitate is produced, but if a *little* ammonia be added, and the mixture stirred with a glass rod, a beautiful crystalline precipitate of acetate of silver ($AgC_2H_3O_2$) is deposited. An excess of ammonia prevents its formation.

* $Fe_2Cl_6 + 6NH_4(C_2H_3O_2) = 6NH_4Cl + Fe_2(C_2H_3O_2)_6$.

Hydrocyanic Acid (HCN).

N.B.—This acid and its soluble salts are extremely poisonous. Even the vapour of the acid is fatal if inhaled in any quantity.

436. (C) Mix a little hydrocyanic acid, or other solution to be tested for cyanogen (CN=Cy) with solution of *potash*, add a little solution of *protosulphate of iron*, then some *perchloride of iron*, and finally an excess of *dilute hydrochloric acid;* a precipitate of Prussian blue is obtained, or if there be very little hydrocyanic acid present, a blue colour is apparent in the solution.*

437. (C) To a little hydrocyanic acid add a few drops of yellow *ammonium sulphide*, and evaporate the solution nearly to dryness in a small porcelain dish (until all odour of the sulphide has disappeared); sulphocyanide of ammonium is formed, and on adding a little perchloride of iron, will give the blood-red colour of ferric sulphocyanide ($Fe_2(CyS)_6$), which is bleached by perchloride of mercury† (434).

438. (C) *Nitrate of silver* added to hydrocyanic acid causes a white precipitate of cyanide of silver (AgCN), which is sparingly soluble in cold dilute nitric acid, but dissolves in the concentrated acid, especially on heating. Cyanide of silver also dissolves readily in ammonia, and is partly precipitated again by the careful addition of excess of nitric acid.

When cyanide of silver is dried, and heated to redness, it evolves cyanogen; if the experiment be performed in a small glass tube closed at one end, the cyanogen may be recognised by its odour, and by its burning with a pink flame. If the cyanide of silver be heated in a crucible, only metallic silver remains, which is entirely dissolved by nitric acid, whilst chloride of silver would remain unchanged.

* The colour varies through different shades of blue and green according to the proportions in which the iron salts are added, but either colour may be taken, if well-marked, as affording evidence of the presence of hydrocyanic acid. $HCy + KHO = KCy + H_2O$. $6KCy + FeSO_4 = K_2SO_4 + K_4(FeCy_6)$.

$$2K_4(FeCy_6) + FeSO_4 + Fe_2Cl_6 = 6KCl + K_2SO_4 + Fe_3(FeCy_6)_2$$

(Prussian blue, or ferroso-ferric-ferrocyanide.) Hydrochloric acid is added to neutralize any excess of potash which would decompose the Prussian blue.

† The analyst must not be misled by a *fugitive* red colour produced by the perchloride of iron with the hyposulphite of ammonia resulting from the oxidation of the sulphide of ammonium by the air.

439. *Dilute sulphuric or hydrochloric acid* decomposes the cyanides (*e.g.*, cyanide of potassium), evolving hydrocyanic acid, which may be recognised by its peculiar odour ($KCN+HCl=KCl+HCN$).

Hydroferrocyanic Acid (H_4FeCy_6).

440. (C) *Perchloride of iron*, added to an acid or neutral solution of a ferrocyanide (*e.g.*, ferrocyanide of potassium), causes a dark blue precipitate of Prussian blue (312).

441. Nitrate of silver gives a white precipitate of ferrocyanide of silver (Ag_4FeCy_6), not dissolved by dilute nitric acid or ammonia; decomposed by heat, the silver being reduced to the metallic state.

Ferrocyanide of silver, allowed to remain in contact with nitric acid, especially if heated, becomes converted into orange-brown ferridcyanide of silver (Ag_3FeCy_6), which is soluble in ammonia.

Tartaric Acid ($H_2C_4H_4O_6$).

442. (C) Heat a small crystal of the acid on platinum foil, or on a slip of glass; it at first fuses, and is afterwards decomposed, with deposition of carbon, and consequent blackening. A peculiar and characteristic odour is at the same time emitted.

443. Heat a small fragment of bitartrate of potash ($KHC_4H_4O_6$) on platinum foil, before the blowpipe or over a spirit lamp. The tartaric acid is thus decomposed, and carbonate of potash is at the same time formed. Place the fragment in a test-tube, and add a few drops of dilute hydrochloric acid, when it will effervesce, showing the presence of carbonic acid.

444. (C) Mix solution of tartaric acid with a drop or two of *ammonia*, still leaving the solution acid. On shaking, a crystalline precipitate of bitartrate of ammonia ($NH_4HC_4H_4O_6$) will be formed. Add enough ammonia to redissolve this, and then *chloride of calcium*. A gelatinous precipitate of tartrate of lime ($CaC_4H_4O_6$) will be formed. Pour part of this into another tube, and test the remainder with *chloride of ammonium*, which will dissolve it.

When the other portion has been set aside for some time, it will have become crystalline, and will then refuse to dissolve in chloride of ammonium, or even in acetic acid, so that it might be mistaken for oxalate of lime, except that after being dissolved in hydrochloric acid, and mixed with chloride of ammonium, it will be found not to be reprecipitated by ammonia (428), unless the solution is briskly stirred with a glass rod, when the tartrate of lime is deposited in a very crystalline state, especially on the lines where the rod has rubbed against the tube. The tartrate of lime precipitated by adding chloride of calcium to the filtered ammoniacal solution of tartar-emetic, does not show the same disposition to become crystalline as is exhibited by that from other tartrates.

445. The tartrate of lime is also soluble, after washing, in a cold solution of potash; if the potash solution be heated, however, the tartrate of lime separates as a bulky precipitate, but redissolves as the solution cools.

446. (C) *Carbonate of potash*, added to tartaric acid, in quantity insufficient to neutralize it, produces a granular crystalline precipitate of bitartrate of potash, the separation of which is much promoted by brisk stirring (356).

447. Acetate of potash with a little free acetic acid will produce the same precipitate in the solution of a neutral tartrate.

448. Tartaric acid and the tartrates, when present in solutions of ferric salts, prevent the precipitation of the hydrate when ammonia or potash is added. This is owing to the formation of double tartrates of iron and the alkali, which are soluble in water, and are not decomposed by an excess of alkali. Tartaric acid also prevents the precipitation of aluminium, manganese, and some other metals, under similar circumstances.

449. When tartaric acid is heated with *strong sulphuric acid*, it blackens at once; if the mouth of the tube be applied to a light, it will be found that carbonic oxide is passing off in small quantity.

449*a*. Potassium permanganate, added to a solution containing tartaric acid mixed with excess of potash, and boiled, gives a brown precipitate of manganic oxide. (Compare 454*a*).

Citric Acid ($H_3C_6H_5O_7$).

450. (C) When heated, citric acid at first melts, and is subsequently decomposed, emitting pungent fumes, which may be distinguished by their smell from those formed by tartaric acid under similar circumstances. A carbonaceous residue remains.

451. (C) *Chloride of calcium*, when added to citric acid which has been neutralized with ammonia, gives a white precipitate of citrate of lime ($Ca_3(C_6H_5O_7)_2$), in the cold if the solution be concentrated, but only on boiling, in a dilute solution, which is insoluble in potash, but soluble in chloride of ammonium.* If this solution be boiled, the citrate of lime reprecipitates. Free citric acid gives no precipitate with this test. Compare (445).

452. (C) *Lime-water*, added in excess, fails to produce a precipitate in a cold solution, but if the mixture be boiled, citrate of lime is thrown down, being less soluble in hot water than in cold.

453. Citric acid and the soluble citrates, when present in solutions containing ferric oxide, aluminium, and some other metals, prevent their precipitation by ammonia, owing to the formation of soluble double salts.

454. Citric acid, when heated with *strong sulphuric acid*, is decomposed; carbonic acid and carbonic oxide are given off with effervescence, and after some time, sulphurous acid is formed, and the mixture becomes dark coloured (449).

454*a*. When tested as in (449*a*), citric acid gives a green solution of potassium manganate (instead of the brown precipitate).

454*b*. $AgNO_3$ added to solution of citric acid, followed by the careful addition of NH_3, yields a white precipitate of argentic citrate, which is dissolved by NH_3 in excess. On heating the solution, the Ag is slowly reduced to the metallic state, whilst, if the same experiment be made with tartaric acid, the reduction takes place much more rapidly.

* The citrate of lime precipitated by boiling is more granular, and is much less easily dissolved by chloride of ammonium and acetic acid than the gelatinous citrate precipitated in the cold.

Benzoic Acid ($HC_7H_5O_2$).

455. (C) When heated in a tube, this acid sublimes, and condenses in the form of beautiful feathery crystals: the vapour has a peculiar aromatic odour, and causes an unpleasant sensation in the throat, inducing coughing.

456. *Perchloride of iron* gives, in neutral solutions, a light yellowish-brown precipitate of ferric benzoate, which is soluble in acids, and is decomposed by ammonia.

457. (C) When the solution of an alkaline benzoate, as benzoate of ammonia, is treated with strong *sulphuric* or *hydrochloric acid*, it is decomposed, and the liberated benzoic acid, being almost insoluble in water, is separated in the form of a white crystalline precipitate.

Gallic Acid ($HC_7H_5O_5$).

458. When heated, gallic acid is charred, and emits a peculiar odour. The acid is sparingly soluble in cold, but dissolves readily in hot water, crystallizing easily on cooling.

459. (C) *Alkalies* mixed with solution of gallic acid cause it to become brown immediately, by absorption of oxygen from the air.

460. (C) *Perchloride of iron* produces a bluish-black precipitate.

Tannic Acid.

461. This acid is also charred when heated. It dissolves readily in cold water.

462. The addition of an alkali causes a solution of tannic acid to become brown when exposed to the air.

463. (C) *Perchloride of iron* produces a bluish-black precipitate.

464. (C) *Dilute sulphuric acid* throws down a white precipitate, which is an insoluble compound of the two acids.*

* The reactions of uric acid will be found in *Bowman's Medical Chemistry.*

CHAPTER VII.

ORGANIC ALKALOIDS.

The organic alkaloids which are most likely to be met with in ordinary analysis are

Morphine (from opium). Strychnine (from *nux vomica*). Quinine and Cinchonine (from cinchona bark).

SECTION I.

Morphine ($C_{17}H_{19}NO_3$).

A solution of the hydrochlorate of morphine may be used.

465. *Concentrated nitric acid* produces, in solutions containing morphine, an orange colour, appearing at once in pretty concentrated solutions, but only after a time, or on boiling, if the solution be dilute.

466. Neutral *perchloride of iron*, with neutral solutions of morphine, gives an inky blue or green colour.

467. *Carbonate of soda* causes the separation of the morphine in the form of a crystalline precipitate, especially if the solution be briskly stirred and allowed to stand; if this be collected on a filter, washed with a few drops of cold water, and moistened with neutral perchloride of iron, the inky colour is very distinct; nitric acid changes it to a bright orange.

(Opium. *Perchloride of iron* produces the fine red colour due to the ferric meconate, which is not bleached by perchloride of mercury (434, 437).

Acetate of lead causes a white precipitate of meconate of lead $Pb_3(C_7HO_7)_2$.

SECTION II.

Quinine (C

A solution of the sulphate of quinine ($(C_{20}H_{24}N_2O_2)_2H_2SO_4$) in dilute sulphuric acid may be used.

468. Acid solutions containing quinine generally exhibit a blue *fluorescence* when viewed in certain lights.

469. *Carbonate of soda* produces a bulky precipitate of hydrated quinine, which shrinks, after a time, and becomes crystalline; if the precipitate be suspended in water, and treated with chlorine, it passes through various shades of violet and red.

470. Strong *chlorine water*, followed by *ammonia*, produces a blue or green colour in solutions of quinine.

SECTION III.

Cinchonine ($C_{20}H_{24}N_2O$).

471. In order to detect the presence of cinchonine in sulphate of quinine, ten grains of the salt are placed in a test-tube with two drachms of washed ether, and twenty drops of solution of ammonia are added. The mixture is agitated violently, and set aside for a minute or two, when it will be found to separate into two layers, the lower one being an aqueous solution of the sulphate of ammonia, and the upper layer an ethereal solution of quinine; cinchonine being insoluble in both, will be seen at the surface of the lower layer.

472. Dissolve cinchonine, or its sulphate, in as little hydrochloric acid as possible, and add ferrocyanide of potassium. A yellow flocculent precipitate is formed, which dissolves when heated with an excess of the ferrocyanide, and is deposited, on cooling, in brilliant yellow crystals, which have a very characteristic appearance under the microscope.

SECTION IV.

Strychnine ($C_{21}H_{22}N_2O_2$).

473. If a minute particle of strychnine be moistened, on a piece of white porcelain, with concentrated *sulphuric acid*, and touched with a drop of solution of *bichromate of potash* upon the end of a glass rod, a deep blue colour will be produced, afterwards changing to violet and red.*

474. A similar change of colour is observed when a drop of concentrated *nitric acid* is poured upon strychnine, and a few particles of the *peroxide* (brown oxide) *of lead* are added.

475. If a solution of strychnine in an acid (hydrochloric) be mixed with *ammonia*, the precipitate which forms at first is redissolved by a slight excess of ammonia, and if the solution be allowed to stand, the strychnine crystallizes in distinct needles.

For the detection of the poisonous alkaloids in mixtures containing organic matter, the student is referred to *Bowman's Medical Chemistry*.

* This test is rendered more delicate by mixing the strong sulphuric acid with a *trace* of the solution of bichromate of potash, and adding a particle of the substance to be tested for strychnine.

PART III.

QUALITATIVE ANALYSIS OF SUBSTANCES, THE COMPOSITION OF WHICH IS UNKNOWN.

CHAPTER I.

476 THE ultimate object of qualitative analysis is, of course, to ascertain the nature of the elements composing any substance presented for examination, without any clue whatever afforded by its origin or history. A system of analysis adapted to this purpose must be so framed that if all the non-metallic and metallic elements were present in the same substance, none should interfere with the detection of the others. Such a method is of necessity somewhat complicated, and would involve a great loss of time if applied to substances containing only one metal and one non-metallic element, or one base and one acid. Cases frequently present themselves in which it may be inferred, with little chance of error, that the substance to be examined is of this description, and the first division of this part of the work will therefore be devoted to the systematic course for the analysis of such substances, which will be found a very necessary introduction to the higher branch of analysis in which the most complex combinations are dealt with.*

* In the Appendix, there will be found a list of salts, &c., which may be taken for practice in qualitative analysis. The student should first examine a few of each kind with the assistance of the book, until he finds himself tolerably familiar with the processes; after which he may try them without reference to the printed directions.

Qualitative Analysis of Substances containing only one Metal and one Non-Metal, or one Base and one Acid.

EXAMINATION OF A SOLID SUBSTANCE.

477 Observe and note down* the appearance of the substance, whether it is **crystalline** or **amorphous**; its **form** and **colour**; especially whether it has any **metallic appearance**.† In the latter case, pass on to (493).

478 PLACE A FRAGMENT OF THE SUBSTANCE IN A SMALL TUBE OF GERMAN GLASS, CLOSED AT ONE END: HEAT IT FIRST OVER A LAMP, AND AFTERWARDS IN THE FLAME OF THE BLOWPIPE.

(*a*) **It undergoes no change, and emits no odour or vapour.**

Absence of *water, organic matter, &c.* Pass on to (479).

(*b*) **It fuses without any other apparent change.**

Probable absence of all metals except *potassium, sodium, barium, strontium, calcium,* and *aluminium.* Absence of *organic matter.* Pass on to (479).

(*c*) **It volatilizes entirely.**

Probable absence of all metals except *ammonium, mercury,* and *arsenic.* The probability is stronger if the volatilization takes place without the aid of the blowpipe. Also absence of *boron* and *silicon.*

(*d*) It emits **brown sulphurous fumes,** condensing in **yellow globules** upon the side of the tube.

Presence of *sulphur.*

* I cannot too strongly insist once more on the importance of making careful and accurate notes of all the experiments and observations which are made; they are not only often absolutely necessary for reference in the subsequent stages of the analysis, but the practice is also of the greatest value to the student in cultivating habits of correct observation and facility of expression; besides at the same time impressing the facts more strongly on his recollection (2).

† In examining minerals and metals, it is advisable to take their specific gravity, when, by reference to a table (such as that in Dr. Thomson's Mineralogy, vol. i. p. 710), we are able to exclude those bodies which differ widely in their specific gravities from the substance under examination.

(*e*) It is **charred,** and emits some **peculiar odour.** Presence of *an organic substance.*

(*f*) **Cyanogen is evolved** (burning with a pink flame). Presence of a *cyanide* or *ferrocyanide.*

(*g*) **Brown nitrous fumes** are evolved. Presence of a *nitrate.**

479 POUR A LITTLE STRONG SULPHURIC ACID UPON THE POWDERED SUBSTANCE IN A TEST-TUBE, and heat gradually, nearly to boiling, testing the odour and combustibility of any gas evolved.

(*a*) **No evolution of gas or vapour** (except that of sulphuric acid); and **no carbonization;** probable absence of all acids except *sulphuric, phosphoric, boracic, silicic, arsenious, arsenic, antimonic,* and *stannic.* Pass on to (480).

(*b*) **Effervescence,** even without heat; probably *carbonic acid* (nearly inodorous), *hydrochloric acid* (pungent fumes), *sulphurous acid* (smell of burning sulphur), *hydrosulphuric acid* (smell of putrid eggs, indicating a sulphide), or *hypochlorous acid* (smell of chlorine). Pass on to (480).

(*c*) **Pungent suffocating acid fumes** are evolved; probably *hydrochloric acid* (indicating a chloride), *hydrofluoric acid* (indicating a fluoride; the tube found to be corroded, after washing and drying),† *nitric acid* (often accompanied by brown vapours).

(*d*) **Violet vapours** are evolved; presence of *iodine.* Pass on to (481).

(*e*) **The substance becomes yellow or red,** and a **yellow chlorous gas** is evolved; presence of *chloric acid* (detonation often occurs on heating). Pass on to (480).

(*f*) **Combustible gas** (carbonic oxide) is evolved; probably *oxalic acid, hydrocyanic acid* (indicating a cyanide), *hydroferrocyanic acid* (indicating a

* The student is reminded that the systematic examination by the blowpipe (165) will be of great service in confirming the results obtained by qualitative analysis.

† If hydrofluoric acid is present, the fumes will probably deposit opaque silica upon a wet glass rod (406).

ferrocyanide; the mixture assumes a blue colour), *citric acid*, or *tartaric acid* (carbonization).

(*g*) Vapours of **acetic acid** are evolved; the substance is an *acetate*. **Benzoic acid** vapours would be known by their odour.

(*h*) **Carbonization takes place;** presence of some *organic substance* other than the acids above named. (*Tartaric*, *gallic*, and *tannic* acids would be carbonized by sulphuric acid.)

480 HEAT A LITTLE OF THE POWDER WITH DILUTE HYDROCHLORIC ACID, observing the odour of the gas, if any, evolved.

(*a*) The gas is **nearly inodorous;** probably *carbonic acid* (effervescence), confirm by (422). Pass on to (481).

(*b*) The gas has the **odour of burning sulphur;** presence of *sulphurous* or *hyposulphurous* acid; confirm by (372) and (375). Pass on to (481).

(*c*) The gas has the **odour of putrid eggs;** presence of a *sulphide;* confirm by (378). Pass on to (481).

(*d*) The gas has the **odour of chlorine;** presence of an *indifferent metallic oxide* (*e.g.*, the peroxides of manganese and lead), of *chloric acid*, of *hypochlorous acid*, or possibly of *nitric acid*. Pass on to (481).

(*e*) An **odour of hydrocyanic acid** is evolved; presence of a *cyanide* or a *ferrocyanide* (blue or green colour).

TO DISSOLVE THE SUBSTANCE FOR ANALYSIS.*

481 Place about ten grains of the powdered substance in a test-tube, and pour upon it two or three fluid drachms of distilled WATER; shake them well together, and if the substance is not dissolved, heat to boiling.

* When the analyst has ascertained how to dissolve the unknown substance, he may often learn much respecting its nature by referring to the Table of Solubilities in the Appendix.

(*a*) The substance is **entirely dissolved.** Pass on to (496).

(*b*) **A great portion** of the substance **is dissolved.** Filter the solution, and examine it by (496).

(*c*) The substance is **not dissolved** to any considerable extent.* Pass on to (482).

482 Boil a fresh portion of the substance (or, if it be scarce, the residue left in (481) with DILUTE HYDROCHLORIC ACID).†

(*a*) It is **entirely dissolved.** Pass on to Table I.

(*b*) **A great portion** of it **is dissolved.** Filter the solution, and examine it by Table I.

(*c*) It is **not dissolved** in any considerable quantity. Pass on to (483).

483 Boil a fresh portion of the substance with DILUTE NITRIC ACID.

(*a*) It is **entirely dissolved.** Pass on to Table I.

(*b*) **A great portion** of it **is dissolved.** Filter the solution and examine it by Table I. Ascertain if the residue presents the characters of *sulphur*, indicating a sulphide (380).

(*c*) It is **not dissolved** to any great extent. Pass on to (484).

484 Boil a fresh portion with STRONG HYDROCHLORIC ACID, and add STRONG NITRIC ACID, very carefully, drop by drop.

(*a*) The substance is **dissolved.** Pass on to Table I.

(*b*) **A great portion** of it **is dissolved.** Dilute the solution with water, filter, and examine it by Table I. Note whether the residue is sulphur (380).

(*c*) The substance is **undissolved.** Pass on to (485).

* If there be any doubt, filter the solution, and evaporate a drop of the clear liquid upon a slip of glass.

† Some substances, especially peroxide of iron, will dissolve only after long digestion with pretty strong hydrochloric acid.

ANALYSIS OF A SINGLE COMPOUND INSOLUBLE IN WATER AND ACIDS.

485 Should the substance remain undissolved after the above course of treatment, heat a small portion of it upon a piece of platinum foil, or of porcelain, or in a porcelain crucible, or in a piece of slightly bent hard glass tube open at both ends.

(*a*) It glows and **burns away,** emitting little or no smell; it is *carbon* (and of course is black).

(*b*) It **fuses** and burns with a **blue flame,** emitting sulphurous acid; it is *sulphur.*

(*c*) It **fuses to a brown drop,** without further change; it is probably *chloride of silver,* and will dissolve when heated with AMMONIA.

(*d*) It **undergoes none of these changes.** Pass on to (486).

486 Examine the substance by (405) to see if it is *fluor spar* (fluoride of calcium).

487 Examine it before a blowpipe, on a platinum wire moistened with hydrochloric acid, to see if it is *sulphate of baryta* (green flame) or *sulphate of strontia* (crimson flame).*

488 Examine it by fusing on charcoal with carbonate of soda, or better, with cyanide of potassium, to see if it is *antimonic acid* (110), the *binoxide of tin* (161), or a compound of *lead* (134).

489 If it be found to contain lead, boil a small portion with ACETATE OF AMMONIA (made by mixing acetic acid with excess of ammonia); if it be *sulphate* or *chloride of lead,* it will be dissolved, and the solution may be tested for sulphuric acid with chloride of barium (370), for chlorine with excess of nitric acid and nitrate of silver (391), and for lead with excess of acetic acid and chromate of potash (201). If undissolved by acetate of ammonia, it is probably *silicate of lead.* Confirm (151).

490 Fuse it with a bead of borax, on platinum wire, to see if it is sesquioxide of chromium (125), or sesquioxide of iron (133).

* Sulphate of lime might also occur here, although it may generally be dissolved by boiling with hydrochloric acid; the analyst must, therefore, not be too hasty in inferring the presence of strontia from the colour of the flame (155).

491 Should the above experiments have failed to convince the analyst of the nature of the insoluble substance, it must be reduced to an impalpable powder (which is best effected in an agate mortar), mixed with three or four times its weight of dry CARBONATE OF SODA (or better, of carbonate of potash and soda), and thoroughly fused, on platinum foil, or in a platinum crucible,* with the aid of the blowpipe, until it remains in a perfectly tranquil state of fusion.†

The fused mass is then allowed to cool, and heated with water for some time (the platinum foil being immersed in the water).

(*a*) The mass is **entirely dissolved** by the water; absence of all metals except *potassium, sodium, aluminium, chromium, manganese, zinc,* and *tin.* Mix the solution with an excess of HYDROCHLORIC ACID, evaporate it to dryness, heat the residue with dilute hydrochloric acid, filter the solution from the separated *silica* (419), and examine it for aluminium, chromium, manganese, zinc, and tin, as in Table I. If none of these be present, a fresh portion of the original substance must be examined for *potassium* and *sodium* by (509).

(*b*) **A portion** of the mass **remains undissolved**; the solution is filtered off (saving the residue for examination by (492), and acidulated with HYDROCHLORIC ACID; one portion being tested for *sulphuric acid* with CHLORIDE OF BARIUM, the other examined as in (*a*).

492 The undissolved residue is washed several times with water, dissolved off the filter in hot DILUTE HYDROCHLORIC ACID, evaporated to dryness; the residue heated with dilute hydrochloric acid, filtered from the separated *silica*, and the solution examined for the metal according to Table I.

* Great care is necessary in using a platinum crucible, that nothing is heated in it which is likely to corrode it. Compounds of the easily reduced and fusible metals, as tin, antimony, lead, bismuth, &c.; substances containing sulphur, as metallic sulphides; caustic alkalies, nitrohydrochloric acid, besides many other substances, are all more or less injurious. When a platinum crucible is heated in a furnace or open fire, it must be placed in a covered earthen crucible to protect it from injury; a little pounded magnesia should be interposed between them, to prevent their sticking together, as at a high temperature the surface of the earthenware is liable to fuse.

† The fusion is more rapidly effected as in (512).

EXAMINATION OF A SIMPLE SUBSTANCE HAVING A METALLIC APPEARANCE.

493 A fragment of the substance is boiled for some time with DILUTE NITRIC ACID.

(*a*) It is **entirely dissolved.** Pass on to Table I.

(*b*) It is **dissolved, with separation of sulphur** (380). Filter the solution, and examine by Table I.

(*c*) A considerable **white residue** is left. This residue is probably either *antimonic acid, binoxide of tin,* or *sulphate of lead.* Pass on to (495).

(*d*) The substance remains **unaltered.** It is probably either *gold, platinum, carbon,* in the state of graphite,* or *iodine* (in which case, **violet vapours** will be evolved). Examine by (494).

494 A small portion of this residue, or of the original substance, is very strongly heated on platinum foil.

(*a*) It **burns slowly away;** *graphite.*

(*b*) It remains **unaltered;** *gold* or *platinum.* Dissolve it in a mixture of hydrochloric and nitric acids, dilute the solution, and add protochloride of tin; gold will give a purple-brown precipitate, and platinum a dark red colour.

495 Boil another fragment of the substance with STRONG HYDROCHLORIC ACID.

(*a*) It rapidly **dissolves, evolving sulphuretted hydrogen** (378). Presence of *sulphide of antimony,* or *sulphide of lead.* Filter the solution and test for lead with dilute sulphuric acid in excess (200), and for antimony by (238). Confirm by blowpipe (110, 134).

(*b*) It **dissolves** slowly with effervescence, but **no** escape of **sulphuretted hydrogen.** Metallic *tin.* Test the solution with PERCHLORIDE OF MERCURY (230).

(*c*) It is **not dissolved** by hydrochloric acid; metallic *antimony.* Add a few drops of STRONG NITRIC ACID. Antimony will entirely dissolve, and may be tested for by (238).

* The graphitoid form of silicon would also remain unaltered.

EXAMINATION OF A LIQUID CONTAINING ONLY ONE METAL AND ONE NON-METAL, OR ONE BASE AND ONE ACID.*

PRELIMINARY EXPERIMENTS.

496 Carefully observe the **colour, taste,** and **smell** of the liquid.

497 Place a drop upon a slip of thin glass and evaporate it to dryness at a gentle heat.

(*a*) **No residue** is left upon the glass; if the liquid is colourless, tasteless, and neutral to test-papers, it is simply water. In any other case, pass on to (498).

(*b*) **A residue** is left; notice its appearance, and the action of heat upon it, making the inferences stated in (478, *c*, *d*, and *e*).

498 Try the action of the liquid upon blue and red litmus paper.

(*a*) The solution is **neutral** or **acid**; pass on to Table I.

(*b*) The solution is **alkaline**; pass on to (499).

499 Mix a little of the alkaline solution, in a test-tube, with enough DILUTE NITRIC ACID to render it distinctly acid. If **no change** is produced, pass on to Table I.

(*a*) **Effervescence** takes place, with evolution of a nearly inodorous gas; presence of an *alkaline carbonate;* pass on to Table VI.

(*b*) The liquid becomes **milky**; if the milkiness does not disappear on adding more nitric acid and gently heating, it is probably sulphur, and indicates the sulphide or hyposulphite of a metal of the third or fourth class. Test for a sulphite with acetate of lead (377), and for a *hyposulphite* with hydrochloric acid (375), in separate fresh portions of the original solution. Examine for the metal by Table I.

(*c*) A **distinct precipitate** separates, which is not dissolved by more nitric acid; this may be *stannic*

* Exclusive of any acid which the analyst may have employed to dissolve the substance under examination.

acid, arsenious acid, antimonic acid, silicic acid, benzoic acid, or *uric acid,* which must have been in combination with an alkali in the original solution. Filter off the precipitate; test the solution for *ammonia* by (360), for *potash* by (354), and for *soda,* after having neutralized with a little potash or carbonate of potash, by (358). Pass on to (500).

500 Mix the original alkaline solution with a considerable quantity of HYDROSULPHURIC ACID, and acidulate it with HYDROCHLORIC ACID.

(*a*) A **yellow precipitate** is produced; filter off the precipitate, and treat with warm CARBONATE OF AMMONIA; if the precipitate dissolves, *arsenious acid* was present; if it does not dissolve, *stannic acid.* Confirm by (252, 161).

(*b*) An **orange precipitate** is produced; presence of *antimonic acid.* Confirm by (110).

(*c*) **No precipitate,** or only a **white precipitate** is produced. Acidulate another portion of the original alkaline solution with HYDROCHLORIC ACID, and add AMMONIA in excess; a **white gelatinous precipitate,** undissolved by the ammonia, is *silicic acid.* Confirm by (419). *Benzoic* and *uric* acids would be dissolved by the ammonia.*

* Benzoic acid may be recognised by its feathery appearance, and by its dissolving readily on boiling the solution in which it was precipitated, emitting its peculiar odour. Uric acid would be identified by mixing the original solution with excess of nitric acid, evaporating just to dryness, and treating the (yellow or red) residue with ammonia, which would develop the pink colour of murexide.

TABLE I.

Analysis of a Neutral or Acid Solution containing a Single Metal.

1.	2.	3.	4.	5.	6.
'o a small part of the solution, add HCl. (If no ppt. *see* Col. 2). Ppt. AgCl Hg_2Cl_2 $PbCl_2$ See Table II.	To the same part add H_2S in excess (*a*). If no ppt. boil the liquid; (if there be still no ppt. See Col. 3). Ppt. *Black or Brown.* HgS, PbS, Bi_2S_3, CuS, SnS. *Yellow*, As_2S_3, SnS_2 *Orange*, Sb_2S_3 See Table III. *White*, S (*b*).	To a fresh part add NH_4Cl (*c*) NH_3 in excess, and $(NH_4)_2S$ (*d*). (If no ppt. see Col. 4). Ppt. *Black.* FeS, CoS, NiS, *Not black*, Al_2O_3, ZnS, Phosphate or oxalate of Ca, Ba, or Sr, Phosphate of Mg, Cr_2O_3, MnS. See Table IV.	To the same part add $(NH_4)_2CO_3$ and heat. (If no ppt. see Col. 5). Ppt. Ca CO_3 Ba CO_3 Sr CO_3 See Table V.	To the same part add Na_2HPO_4 and stir. Ppt. indicates Mg.	Test for K, Na and NH_4 as in Table VI.

TABLE II.

Analysis of the Precipitate Produced by HCl (*e*).

(Add a little more HCl, to make sure that the precipitate will not re-dissolve). Pour off the liquid, and shake the precipitate with NH_3.

Dissolved. AgCl. Add dil. HNO_3 in excess Precipitate AgCl, indicating Ag (*f*).	Blackened. Hg_2Cl_2 Presence of Hg as a mercurous compound. Confirm by (209).	Unchanged. $PbCl_2$ Presence of Pb. Confirm by (199), (200), (201).

TABLE III.

Analysis of the Precipitate produced by H_2S.

<table>
<tr>
<td colspan="8">1. The precipitate is black or brown.
Test separate portions of the original solution with</td>
<td colspan="3">2. The precipitate is yellow or orange.
Collect it on a filter; wash it, and pour warm $(NH_4)_2CO_3$ over it.</td>
</tr>
<tr>
<td rowspan="3">1.
Dil. H_2SO_4
white ppt.
$Pb SO_4$
indicates
Pb.
Confirm by
(201, 202).</td>
<td colspan="4">2.
NH_3.</td>
<td rowspan="3">3.
KHO
in excess
yellow
ppt.
indicates
Hg (g)
(218).</td>
<td rowspan="3">4.
HCl
and
Cu
Boil
Silvery
coating
Hg (g)
(218).</td>
<td rowspan="3">5.
Hg Cl_2
White
or
Grey
ppt.
indicates
Sn (h)
(229).</td>
<td rowspan="3">Solution.
Add HCl
in excess
(If no ppt.
see next
column).
Yellow ppt.
indicates
As (i)
(267).</td>
<td colspan="2" rowspan="2">Undissolved.
(Orange, Sb_2S_3, Yellow, SnS_2).
Add to the original solution NH_3 in slight excess, and HCl in slight excess.

Introduce a piece of Zinc and set aside for some minutes.</td>
</tr>
<tr>
<td rowspan="2">Rich blue
solution
indicates
Cu.
Confirm
by
(226).</td>
<td colspan="3">White ppt. Dissolve in very little dil. HCl and divide into three parts.</td>
</tr>
<tr>
<td>1.
Add
much
Water,
milky
ppt.
indicates
Li.
Confirm
by (222).</td>
<td>2.
Add
KI
(Red ppt.
indicates
Hg)
in excess.
Brown-
yellow
solution
indicates
Bi
(222).</td>
<td>3.
Introduce
a few
pieces of
bright
Copper
and boil.
Silvery
coating
indicates
Hg (g)
(218).</td>
<td>Black
ppt.
Sb
(236).</td>
<td>Grey ppt. Sn.
Pour off the
liquid.
Boil the ppt. with
Conc. HCl, and
test the solution
with $HgCl_2$.
White or Grey
ppt.
indicates
Sn (k).</td>
</tr>
</table>

TABLE IV.

Analysis of the Precipitate produced by $(NH_4)_2S$.

<table>
<tr><td colspan="3">1. The precipitate is black.
Treat a little of the ppt. with dil. HCl.</td><td colspan="8">2. The precipitate is not black (m).
Test separate portions of the original solution (n) with</td></tr>
<tr><td rowspan="4">Dissolved or changed to white (sulphur). Presence of Fe (l).</td><td colspan="2">Undissolved.
Test the original solution with KHO in excess.</td><td colspan="2">1.
NH_3 in excess.</td><td colspan="5">2.
KHO in excess.</td><td>3.</td></tr>
<tr><td rowspan="3">Blue ppt. Presence of Co (327, 329).</td><td rowspan="3">Pale-green ppt. Presence of Ni (317, 318).</td><td rowspan="3">Precipitate See Col. 2.</td><td rowspan="3">Solution. Add a drop of $(NH_4)_2S$ White ppt. indicates Zn (292). Pink ppt. indicates Mn (298).</td><td rowspan="3">Green solution indicates Cr (283).</td><td colspan="3">Colourless solution.
Add Acetic Acid in excess.</td><td rowspan="3">Precipitate becoming brown when shaken with air indicates Mn (298).
Precipitate remaining White. See Col. 3.</td><td rowspan="3">NH_3 in slight excess and Acetic Acid in excess. Ppt. See note (o).
—
If no ppt. See note (p).</td></tr>
<tr><td rowspan="2">Ppt. indicates $AlPO_4$.</td><td colspan="2">Solution.
Add NH_3 in excess.</td></tr>
<tr><td>Ppt. indicates Al.</td><td>Solution. Add $(NH_4)_2S$ Ppt. indicates Zn.</td></tr>
</table>

TABLE V.

Analysis of the Precipitate produced by $(NH_4)_2CO_3$.

Collect the precipitate upon a filter, wash it, and dissolve it in a very little dil. HCl.

(In most cases, the following tests may be applied to the original solution, instead of the dissolved precipitate.)

1.	2.	3.
Add, to a small portion, *dil.* H_2SO_4. If no precipitate, Ba and Sr are absent. See Col. 3. If a precipitate is produced (*q*). See Col. 2.	To a fresh portion Add $CaSO_4$. Immediate precipitate indicates Ba. Precipitate on standing or heating indicates Sr. (See note *r*.)	To a fresh portion Add NH_3 in excess and (Ammonium oxalate) $(NH_4)_2C_2O_4$. Precipitate indicates Ca. (341).

TABLE VI.

Detection of Potassium, Sodium, and Ammonium.

1.	2.	3.
Boil a portion of the *original substance* with KHO. Odour of NH_3 indicates presence of NH_4. (361, 363).	Make a rather strong solution of the substance, and stir it with Tartaric Acid $(H_2C_4H_4O_6)$ or with $PtCl_4$ upon a slip of glass. Precipitate (usually on the lines of friction) indicates K (149).	Take a little of the substance upon a clean Pt wire, and expose in a non-illuminating flame. Strong *Yellow* colour indicates Na. (Confirm by 358).

TABLE VII.

Analysis of the Solution of a Simple Substance for the Acid or Non-metallic Body.

Case 1.—*The substance has been dissolved in water and the solution is not strongly acid.*
(This may be inferred if it does not effervesce on adding a little sodium carbonate.)

Part 1. Add Ba $(NO_3)_2$	Part 2. Add $AgNO_3$.	Part 3. Add $CaCl_2$ (*t*) Precipitate:	Part 4. Add Fe_2Cl_6 (*t*). Precipitate:	Part 5. Test for a *nitrate*	Part 6. The following acids may still have escaped detection, and should be specially tested for.
White ppt. *insoluble* in *dil.* HNO_3 indicates a *sulphate*. White ppt. *soluble* in *dil.* HNO_3; with *effervescence* indicates a *carbonate*, without effervescence See Part 3. *Yellow* ppt. soluble in *dil.* HNO_3 indicates a *chromate* (288, 289).	White ppt. *insoluble* in *boiling* HNO_3 indicates a *chloride* (392, 393). White ppt. *insoluble* in *cold* HNO_3 but *soluble on boiling*, indicates a *cyanide* (436). White ppt. changed to *brown* by boiling HNO_3 indicates a *ferrocyanide* (440). *Yellow* ppt. *insoluble* in HNO_3 and in NH_3 indicates an *iodide* (399). *Black* ppt. indicates a *sulphide* (377) (*s*). White ppt. changing to yellow, brown, and black indicates a *hyposulphite* (375). White ppt. becoming brown when boiled, indicates a *sulphite* (372) or a *borate* (411).	*Insoluble* in acetic acid, indicates an *oxalate* (427), or a *fluoride* (405), or a *silicate* (419); *Soluble* in acetic acid; add a drop of Fe_2Cl_6. White ppt. indicates a *phosphate* (382) (*u*). A ppt. by $CaCl_2$ *soluble* in acetic acid, may also be caused by a *borate* (411), or a *tartrate* (444). If no ppt. with $CaCl_2$, boil the solution to test for *citric acid* (451).	Ferric *phosphate* (white), *arseniate* (white) (*u*), *sulphide* (black), *ferrocyanide* (blue), *gallate* (blue-black), *tannate* (blue-black), *benzoate* (buff) (*v*). Colour: *Sulphocyanide* (dark red) (434) *Acetate* (light red) (432) *ferricyanide* (dark-brown) (441) *gallate* or *tannate* (inky blue), *sulphite* or *hyposulphite* (transient red or purple).	by adding $FeSO_4$ and, very carefully, conc. H_2SO_4 Brown band (366, 367).	*Chloric* (394) *Hypochlorous* (397) *Boracic* (411, 412) *Acetic* (432).

TABLE VIII.

Analysis of the Solution of a Simple Substance for the Acid or Non-metallic Body.

Case 2.—*The substance has been dissolved in water, and the solution is strongly acid.*

Part 1.	Part 2.	Part 3.	Part 4.		Part 5.
Add $Ba(NO_3)_2$ White ppt. *insoluble* in *dil.* HNO_3 indicates a *sulphate.*	Add $AgNO_3$ Precipitate. See Part 2 in Table VII.	Test for HNO_3 with $FeSO_4$ and conc. H_2SO_4 Brown band (366, 377).	Add Na_2CO_3 till the solution is strongly alkaline, boil for a minute or two, filter from any precipitate (containing the metal), acidulate slightly with HNO_3, heat gently to expel CO_2. Add NH_3 in slight excess, and divide into two parts.		Test specially for *Sulphurous* (372) *Acetic* (432) *Chloric* (394 et seq.)
			To one part add $CaCl_2$ Precipitate. See Part 3 in Table VII.	Evaporate the other part till it is neutral, and add Fe_2Cl_6 drop by drop. See Part 4 in Table VII.	

TABLE IX.

Analysis of a Simple Substance for the Acid or Non-metallic Body.

Case 3.—*The substance is insoluble in water, but dissolves in acids.**

1.	2.	3. Boil a portion of the substance with Na_2CO_3 and a little water for a minute or two, filter, and divide into six parts.					
Boil a portion of the substance with HCl. *Effervescence* indicates a *carbonate* (422) or a *sulphide* (378) Filter, if necessary, and add $Ba(NO_3)_2$ Ppt. indicates a *sulphate.*	Boil a portion of the substance with HNO_3. *Spongy flakes* of sulphur indicate a *sulphide.* *Violet-vapour* indicates an *iodide.* Filter, if necessary, and add $AgNO_3$. Ppt. indicates a *chloride.*	Part 1. Add $Ba(NO_3)_2$ Ppt. may be due to the excess of Na_2CO_3 only. See Part 1 in Table VII.	Part 2. Add Acetic Acid in excess, and Fe_2Cl_6. White precipitate indicates a *phosphate* (388) *Blue* precipitate indicates a *ferrocyanide.* *Dark-brown colour* indicates a *ferricyanide.*	Part 3. Add excess of HCl and observe any indication of a *sulphite* (371, 372), a *sulphide* (378), or a *hyposulphite* (375).	Part 4. Add $FeSO_4$, Fe_2Cl_6, and excess of HCl. *Blue* ppt. indicates a *cyanide.*	Part 5. Add *dil.* H_2SO_4 in slight excess; $FeSO_4$ and very carefully conc. H_2SO_4 Brown ring indicates a *nitrate.*	Part 6. Neutralise exactly with HNO_3 and add Fe_2Cl_6 Dark-red colour indicates a *sulphocyanide.*

* A substance insoluble in acids must be examined as in (485).

Notes to Table I.

a. Be sure that the Hydrosulphuric Acid has a strong smell of the gas, since it becomes useless when kept (641). To ascertain if it is in excess, close the mouth of the tube with the thumb, and shake violently; it should still smell strongly of the gas. If much nitric acid has been used in dissolving the substance, it is advisable to evaporate down and dilute with water, before adding hydrosulphuric acid.

b. A white precipitate of sulphur (appearing as a bluish milkiness in the liquid) is caused by the presence of some substance capable of removing the H of the H_2S; such substances are ferric salts, nitrous acid, chlorine, sulphurous acid, chloric acid, chromic, manganic, and permanganic acids. Arsenic acid also causes separation of sulphur, followed by precipitation of yellow As_2S_3.

c. Ammonium chloride is added to prevent the precipitation of magnesium hydrate by ammonia (345); the volume of solution of ammonium chloride added should be about twice that of the liquid.

d. Ammonia must be added till the liquid smells of it after closing the tube with the thumb and shaking it. Any free acid would decompose the ammonium sulphide, precipitating sulphur.

Notes to Table II.

e. Sulphur is sometimes precipitated here. It would be known by its not separating in distinct particles, and by the attendant odour of SO_2 (from a hyposulphite).

f. The presence of silver may be confirmed by adding potash in excess to the original solution; a brown precipitate of Ag_2O would be produced.

Ferricyanide of potassium produces a red-brown precipitate in solutions of silver.

Notes to Table III.

g. The mercury detected here is present as a mercuric salt. If nitric acid was used in dissolving the original

substance, it would convert a mercurous salt into the mercuric form. Mercurous salts are always blackened when treated with potash.

h. The tin detected here is present as a stannous salt. Gold and platinum would also give a black or brown precipitate with H_2S. The former would be precipitated as a purplish powder (from the original solution) by protosulphate of iron. Platinum would give a yellow precipitate with chloride of ammonium.

i. This arsenic may have been present in the original substance as arsenious or arsenic acid (273). If nitric acid has been used in dissolving the substance, any arsenious acid will have become converted into arsenic acid.

k. The tin detected here is present as a stannic compound. If nitric acid has been used in dissolving the substance, it would have converted a stannous into a stannic compound.

Cadmium gives a yellow precipitate with H_2S, distinguished from the sulphides of arsenic and tin by its insolubility in ammonium sulphide.

NOTES TO TABLE IV.

l. Confirm the presence of iron, and ascertain whether it is present as a ferrous or ferric salt by (306, 312). If nitric acid was used in dissolving the substance, any ferrous salt would have been converted into a ferric salt.

m. This precipitate sometimes has a green colour, due to the presence of a very little iron as an impurity.

Should ammonia produce a bright yellow precipitate, without adding ammonium sulphide, it is probably barium chromate.

n. Should this solution have a fine *red* colour, it probably contains a permanganate (p. 90, *note*), or an acid chromate (287). If it be *yellow*, it may contain a neutral chromate (287). If *green*, probably a manganate (p. 90, *note*), or a salt of chromic oxide (283). The basylous metal present in a chromate, manganate, or permanganate must be detected as in the analysis of complex bodies.

o. This precipitate, if undissolved by excess of acetic

acid, is probably calcium oxalate (429, 430). It might also possibly be barium oxalate or strontium oxalate. The precipitate, or the original substance, should be heated (429) until converted into a carbonate, which may then be analysed by Table V.

A semi-transparent gelatinous precipitate by ammonia, insoluble in acetic acid, is probably calcium fluoride (405).

p. If the precipitate produced by ammonia be soluble in acetic acid, it is probably a phosphate of Ca, Ba, Sr, or Mg. Add to the acetic solution a drop of ferric chloride; a white ppt. indicates a phosphate; add more ferric chloride till the solution is reddish, boil and filter. The filtered liquid, which should be colourless, may be examined for the metals as usual. (See Table I. col. 4.)

Notes to Table V.

q. A precipitate here does not certainly indicate Ba or Sr, since Ca may be precipitated as $CaSO_4$ in strong solutions.

r. If the solution is acid, Sr may escape detection here. In that case, evaporate the acid solution to dryness, and dissolve the residue in water before testing with $CaSO_4$.

Notes to Table VII.

s. A solution containing a free alkali or alkaline earth would give a nearly black precipitate of silver oxide.

t. If silver, lead or (mercurous) mercury be present, this test must be omitted.

u. An arseniate might be mistaken for a phosphate here; but arseniates give a brown-red precipitate with silver nitrate, with which phosphates give a yellow precipitate.

v. If the original solution was alkaline, a red-brown precipitate of ferric hydrate may be produced here.

CHAPTER II.

QUALITATIVE ANALYSIS OF COMPLEX SUBSTANCES WHICH MAY CONTAIN TWO OR MORE METALS AND NON-METALS, OR TWO OR MORE BASES AND ACIDS.

Introductory Remarks.

501. Unless we have reason to know that a substance intended for analysis contains only one base and one acid, it is necessary to assume that it *may* contain any or all of the more common saline compounds. Such an analysis is of course considerably more complicated than that of a single salt; and consequently the necessity of having a well-devised scheme of experiments is here even greater than in the former case, when only one base and acid had to be detected.

The student must be careful when analysing complex substances, that he adds sufficient of the various reagents, to throw down *the whole* of the metals affected by them, since any traces of the metals belonging to a class supposed to have been entirely removed from the solution, would materially interfere with the indications afforded by the subsequent tests. For example, in the analysis of a mixture of a salt of lead and a salt of lime, if sufficient hydrosulphuric acid were not passed through the solution to separate the whole of the lead, a black precipitate of sulphide of lead would be formed on the addition of hydrosulphate of ammonia to the filtered liquid, indicating the presence of one or more metals of the third class, none of which are really present. On the other hand, the addition of a large excess of any of the reagents is also to be avoided, as being not only useless and wasteful, but in many cases mischievous.

Both these errors may be avoided by adding the reagents in small successive portions: and when the experimenter has reason to think that he has added sufficient, let him filter a few drops of the mixture, and apply to the solution a little more of the reagent: if this produces no

further precipitate, he may conclude that enough has been added.

In the course of an analysis, especially of a complicated substance, it is often necessary to have several solutions in hand at the same time; to avoid confusion, each of these should be labelled with a bit of gummed paper, with a letter or mark upon it, referring to a corresponding letter in the note-book (2).

As one portion of the substance to be analysed has to be carried through several operations, it is advisable that the quantity operated on should not be very small. When the substance is a solid, twenty or thirty grains may be used; and when in solution, an ounce or two (according to the degree of concentration) will be found a convenient quantity.

Where it is possible, the substance should be weighed previously to analysis, and carried through the whole process without being divided, in order that some idea may be formed of the relative proportions of the substances detected.

EXAMINATION OF A COMPLEX SOLID SUBSTANCE.

502 The substance should first be examined according to the directions given in (477–480), but the analyst must be more careful in drawing his conclusions from the indications presented, on account of the complication of phenomena arising from the presence of more than one compound.

The process of dissolving the substance also requires modification.

TO DISSOLVE THE SUBSTANCE FOR ANALYSIS.

If the substance has a *metallic appearance*, pass on to (513).

503 Boil about 20 grains of the powdered substance, in a test-tube or small flask, with two or three fluid drachms of distilled water for a minute or two.

(*a*) **The substance is entirely dissolved.** Pass on to (517).

(*b*) **An insoluble residue is left.** Set the tube aside for a few minutes, to allow the residue to settle. Pour the liquid upon a filter (leaving the residue in the tube, if possible), and reserve the solution for examination according to Table X. Wash the residue once or twice with water, by decantation, if possible, or upon the filter (not allowing the washing waters to mix with the solution first filtered), and proceed as in (504).*

504 Boil the residue in a tube or flask, with about a drachm of STRONG HYDROCHLORIC ACID (unless *silver* or *lead* be suspected,† when nitric acid must be employed) for a minute or two,‡ add about an equal quantity of water, and again boil.

(*a*) **The substance is entirely dissolved.** Pass on to Table X.

(*b*) **An insoluble residue is left.** Add a few drops of STRONG NITRIC ACID (or of hydrochloric if nitric acid was employed before), and again boil. Should this fail to dissolve it, add more water, boil, and filter,§ reserving the clear solution for examination by Table X. Wash the residue upon the filter as long as the washings are acid to litmus paper, dry it, and treat it according to (505).

* If the residue has been collected upon a filter, it may either be washed through the perforated filter with as little water as possible, or it may be dried and carefully detached.

† From the preliminary blowpipe examination.

‡ If red oxide of iron be present, prolonged digestion with hydrochloric acid is often necessary.

§ Should any gelatinous silica have separated, as is the case when slags, cements, and zeolitic minerals are decomposed by acids, it will be advisable to evaporate the mixture to dryness, and to treat the dry residue with hydrochloric acid so as to leave all the silica undissolved. The liquid may then be filtered.

ANALYSIS OF A COMPLEX COMPOUND, INSOLUBLE IN WATER AND ACIDS.*

505 (*a*) **The insoluble substance is black.** Heat a small portion of it upon a piece of platinum foil; if it burns entirely away, it contains nothing but *carbon* and perhaps *sulphur*, which may be recognised by its odour; but if any incombustible residue be left, the substance must be examined according to (506).

(*b*) **The insoluble substance is not black.** Heat a small portion upon a piece of platinum foil, to ascertain whether it contains any *sulphur;* if any incombustible residue be left, examine the original insoluble substance according to (506).

506 Fuse a small portion of the insoluble substance on charcoal, with carbonate of soda, before the blowpipe (166) and examine very carefully, to see if it contains any *lead*, *tin*, *silver*, or other easily reducible metal capable of corroding platinum.

(*a*) Indications of *reduced metal* are obtained. Pass on to (510).

(*b*) *No metal* is obtained. Examine as in (507).

507 Reduce the insoluble substance to a very fine powder,† and reserve one-fourth of it for examination by (509). Mix the remaining three-fourths with about four times its weight of CARBONATE OF SODA (or better, of the carbonate of potash and soda) and fuse it‡ in a platinum crucible, or capsule, or upon a piece of platinum foil, over a blowpipe-flame, retaining the mass in a fused state for several minutes. After cooling, place the crucible and its contents in water, and digest, at a moderate heat, with occasional stirring, until the fused mass is entirely detached from the crucible, which may then be removed. When the fused mass has been thoroughly disintegrated,

* The chief substances which would remain undissolved after the above treatment are the *sulphates of baryta, strontia* (and possibly of *lime* and *oxide of lead*), *chloride of silver, silicic acid*, and the *silicates* of any of the *metallic oxides; alumina* and *oxide of chromium* in certain states, *fluoride of calcium, binoxide of tin, antimonic acid, chrome-iron ore, sulphur, charcoal.*

† Should any carbon or sulphur have been discovered in it (505) the substance should be calcined in an open crucible until these are burnt off.

‡ See note to (491).

filter the solution, and examine it as in (508). The residue on the filter is well washed until the washings are no longer precipitated by chloride of barium, some hot DILUTE HYDROCHLORIC ACID is poured over it, and should this fail to dissolve it, it should be rinsed into a tube and boiled with the acid.

(*a*) The residue is **entirely dissolved.** Evaporate the solution to dryness (to convert the silica into the insoluble form), and heat the residue with DILUTE HYDROCHLORIC ACID. Filter the solution from any *silica*,* and examine it for metals according to Table X. except for potassium and sodium, which must be sought for by the method described at (509).

(*b*) An **insoluble residue** is left. This should be washed once or twice, and boiled with STRONG HYDROCHLORIC ACID (the hydrochloric solution being treated as in (*a*),) and should this fail to dissolve it, the residue, which consists of some substance (such as chrome-iron ore), not attacked by the alkaline carbonates, should be dried and fused with a mixture of equal weights of carbonate of soda and nitrate of potash, on platinum foil. On treating the mass with water, the solution, which is yellow if chromium be present, may be examined as in (508*a*), a part being reserved for examination for acids by (Table XVII.). The residue, after washing, may be digested with strong hydrochloric acid, which will now at least partly dissolve it,† and the solution may be examined as in (*a*).

508 The solution obtained by digesting the original fused mass from (507) in water is divided into two parts, to be examined according to *a* and *b*.

* The silica may be identified by its easy solubility in hot solution of potash. Titanic acid is occasionally left behind with the silica. It may be detected by fusing the dried silica with bisulphate of potash, and treating the fused mass with water, which will dissolve the titanic acid. On filtering the solution, largely diluting it with water, and boiling for an hour, the titanic acid will give a milky precipitate, which may be collected and identified by fusing with a bead of microcosmic salt in a good reducing flame, when a pearly blue bead will be obtained. Titanic acid has a remarkable tendency to adhere firmly to the glass vessels.

† Any portion still remaining insoluble must be tested before the blowpipe (165, &c.).

(*a*) One portion is acidulated with HYDROCHLORIC ACID and evaporated to dryness, to render the silica insoluble. The residue is warmed with dilute hydrochloric acid, any *silica* being filtered off, and examined (see note to 507*a*), and the solution tested for metals (Table X.), especially for *aluminium*, *chromium*, *tin*, and *zinc*, which may exist in alkaline solutions.

(*b*) The other portion of the solution may be examined for acids by (Table XVII.), though it is seldom necessary to test insoluble substances for other than *sulphuric* (370), *silicic* (419), *hydrochloric* ([illegible]), and *hydrofluoric* (406, 409).

EXAMINATION OF INSOLUBLE COMPOUNDS FOR POTASSIUM AND SODIUM.*

509 The finely powdered substance is intimately mixed with one part by weight of FLOWERS OF SULPHUR, and six parts of NITRATE OF BARYTA; the mixture is heated in a porcelain crucible over a spirit-lamp, or air-gas flame, until the deflagration is over. After cooling, the fused mass is detached with a knife, finely powdered, thrown into boiling water, in a dish, boiled for a minute or two, and filtered from the sulphate and silicate of baryta, &c., which may remain. The filtrate is mixed with ammonia and carbonate of ammonia, to separate baryta and other alkaline earths which may be present, and after these have been filtered off, the solution is evaporated to dryness, the dry residue calcined as long as any fumes are evolved, and further treated as in (Table XVI.).†

510 If any metal was reduced in the experiment with the blowpipe (506), examine it carefully to see if any certain inference can be drawn from its malleability, &c., and proceed to examine the rest of the insoluble substance according to (511).

* This is not generally necessary unless silicic acid or hydrofluoric acid has been found in the previous examination.

† This process will generally be found more convenient than the older method of fusing with hydrate of baryta.

511 Boil the insoluble substance with a strong solution of ACETATE OF AMMONIA (prepared by mixing acetic acid with ammonia in *excess*) for a few minutes.

(*a*) It is **entirely dissolved,** and consisted therefore of *sulphate of lead* or *chloride of silver*. Add to the solution a slight excess of nitric acid, which will precipitate the chloride of silver, and test for lead, in the filtered solution, with hydrosulphuric acid.

(*b*) **A residue is left;** filter the solution, and test it as in (*a*), for sulphate of lead and chloride of silver. Wash the residue and treat it as in (512).

512 Heat a *small portion* of the residue on platinum foil.

(*a*) **It burns entirely away;** and consists therefore either of *sulphur* (known by the odour), or of *carbon* (known by the colour), or of both.

(*b*) An **incombustible residue** is left. Dry the whole of the insoluble residue from (511*b*), and heat it to dull redness in a porcelain crucible until all the sulphur and carbon, if any be present, are burnt off: then carefully examine a part of the residue on charcoal, with carbonate of soda, to see if it still contains any reducible metal. If no metal be found, examine it as in (507), but otherwise, proceed as follows:—

Mix the finely powdered residue with twice its weight of dry CARBONATE OF SODA, twice its weight of finely powdered CHARCOAL, and twelve times its weight of dry NITRATE OF POTASH. Place the mixture in a perfectly clean iron tray, or a thin porcelain dish, and kindle it in the centre with a lighted match.*

When the fused mass is cool, boil it with water in a porcelain dish, with occasional stirring, until it is entirely disintegrated, when it may be filtered off. Examine the filtered solution as in (508).

The residue on the filter is well washed till the washings are no longer alkaline, some hot DILUTE NITRIC ACID is poured over it, and should this fail to dissolve it, it should be rinsed into a tube and boiled with the acid.

* Another method consists in fusing the substance with carbonate of soda or carbonate of potash and soda as in (507) using a porcelain instead of a platinum crucible, but the porcelain is generally corroded, thus necessitatin g a separate examination for silica and alumina.

(*a*) The residue is **entirely dissolved.** Evaporate the solution to dryness, to render silica insoluble, heat the residue with DILUTE NITRIC ACID, filter from any *silica* (see note to 507*a*) and examine the solution for metals as in (Table X.), except for potassium and sodium, which must be sought as in (509).

(*b*) An **insoluble residue** is left. Filter this off, and examine the solution as in (*a*). Examine the residue according to (507*b*).

EXAMINATION OF A COMPLEX SUBSTANCE HAVING A METALLIC APPEARANCE.

The substance is reduced to as fine a state of division as possible, and about 10 grains of it are boiled with DILUTE NITRIC ACID, in a flask or large test-tube, as long as any action appears to take place; about an equal quantity of WATER is then added, and the mixture again boiled.

(*a*) The substance is **entirely dissolved.** Pass on to (Table X.).

(*b*) **A residue is left;** the solution is filtered or poured off from the residue, and examined according to Table X. Examine the residue as in (514).

514 The residue is well washed, dried, and calcined in a porcelain crucible at a moderate heat, any indication of the presence of *sulphur* being carefully looked for.

(*a*) **The substance burns entirely away.** It consists of *sulphur* only.

(*b*) An **incombustible residue** is left. If this residue be **light-coloured,** it may contain *binoxide of tin*, *antimonic acid*, or *sulphate of lead*, and should be examined by (516). If the residue be **dark-coloured,** it may contain, in addition to those substances, *gold*, *platinum*, and carbon in the state of graphite. Examine it as in (515).

515 Boil this dark-coloured residue with a mixture of HYDROCHLORIC ACID with one-third of NITRIC ACID.

(*a*) It is **entirely dissolved,** and therefore probably

consisted of finely divided *gold* or *platinum*, or both. Test one part of the solution for gold with PROTOCHLORIDE OF TIN (494*b*), and another for platinum with chloride of ammonium.*

(*b*) An **insoluble residue** is left. This may consist of *sulphate of lead, antimonic acid, binoxide of tin, arsenic acid* (all **white**), and *carbon* as graphite.† If the residue is **black,** heat it to redness for some minutes upon a strip of platinum foil; if it burns entirely away, it is simply carbon; but should it be white, or leave an incombustible residue, treat it as follows:—

516 Boil with a strong solution of ACETATE OF AMMONIA (acetic acid mixed with ammonia in slight excess) for a few minutes.

(*a*) The substance is **entirely dissolved;** test the solution for *sulphate of lead* as in (489).

(*b*) **A residue is left.** Filter the solution, and test it for lead (201). Dry the washed residue and mix it in a porcelain crucible with three or four parts of powdered CYANIDE OF POTASSIUM. Cover the crucible, heat it strongly till the contents are in a state of perfect fusion, and keep it so for ten minutes. (Observe whether there is any garlic odour, indicating *arsenic*). When the crucible is cool, fill it with boiling water, and stir well, in order to dissolve the cyanate of potash and cyanide of potassium; allow the undissolved portion (possibly tin, antimony, arsenic, and carbon) to subside, wash it by decantation,‡ rinse it into a test-tube, and boil with a little STRONG HYDROCHLORIC ACID for a few seconds. Decant a little of the hydrochloric solution into another tube, and test for *Tin* with PERCHLORIDE OF MERCURY (230). To the rest of the acid (on the residue) add a drop or two of NITRIC ACID, and continue to

* Some sulphate of lead would also be dissolved by the mixed acids, but need not be regarded.

† Some of the rarer metals, such as the alloy of osmium and iridium, might also be expected here, as might also titanic acid. Although arsenic acid is very soluble when alone, it may occur here in the form of an insoluble compound with binoxide of tin.

‡ If there be any metallic globules, try them as to their malleability. If they are brittle, it is well to pound them in a mortar before boiling with hydrochloric acid.

boil. Any *Carbon* will remain undissolved. The acid solution is mixed with an excess of saturated HYDROSULPHURIC ACID: the precipitate collected upon a filter, and examined as directed in Table XII. (Solution C 1). The filtered solution is mixed with more hydrosulphuric acid and boiled, when the formation of a yellow precipitate indicates *Arsenic*.

EXAMINATION OF A LIQUID SUPPOSED TO CONTAIN TWO OR MORE METALS AND NON-METALS, OR BASES AND ACIDS.*

517 Examine the liquid as directed in (496, 497, and 498).

(*a*) The solution is **neutral or acid**; pass on to Table X.

(*b*) The solution is **alkaline**; pass on to (518).

518 Mix a part of the alkaline solution with enough DILUTE NITRIC ACID to render it distinctly acid. If **no change** is produced, pass on to Table X.

(*a*) **Effervescence** takes place, with evolution of a nearly inodorous gas; presence of an alkaline carbonate; pass on to Table X.

(*b*) The liquid becomes **milky**; if the milkiness does not disappear on adding more nitric acid, and gently heating, it is probably sulphur, and indicates the presence of a *sulphide* or *hyposulphite*; boil, filter, and pass on to Table X.

(*c*) A distinct **precipitate** separates, which is not dissolved by more nitric acid. Filter off the preci-

* When a complex compound has been partly dissolved by water, and partly by acids, it becomes a question whether the solutions should be mixed and examined together. In general, it may be stated that when a knowledge of the arrangement of the constituents is desired, the solutions should be examined separately, but when the sole object is to learn what metals and non-metallic bodies are present, the solutions may be mixed, observing these precautions before mixing:—(1) Acidulate the aqueous solution with nitric acid, and examine any precipitate as in (518*c*); afterwards mix the clear liquid with hydrochloric acid, and reserve any precipitate to be examined by Table XI. (2) Mix the nitric solution with hydrochloric acid, and examine any precipitate by Table XI. (3) If, on mixing the acid solutions after these precautions, any precipitate be obtained, it must be collected and examined as an insoluble substance (506).

pitate and examine the solution by Table X. Wash the precipitate, and endeavour to dissolve it by boiling with strong hydrochloric acid and a few drops of nitric acid; filter the solution, and examine it by Table X. If there be any undissolved residue, it must be washed, dried, and examined according to (505).

TABLE X.

Analysis of a Solution (A) which may contain all the Common Metals.

(Reserve a small portion.) Add dil. HCl in slight excess. If a precipitate is produced, heat gently, and if it is not redissolved, filter.

<table>
<tr><td>Precipitate B.
Wash, and examine as directed at Table XI.</td><td colspan="3">Solution B (a). Test a small portion with H_2S, and boil. If there be no precipitate, or only a white precipitate (S), treat the rest of the liquid as Solution C, omitting the directions in parentheses. But should H_2S cause a precipitate, mix the whole of the liquid with a considerable volume of H_2S water, and, unless it now smells very strongly of H_2S, pass H_2S gas through it, until it does so, and retains the smell after stirring. Filter off the precipitate.</td></tr>
<tr><td>Precipitate C.
Wash, and examine as directed at Table XII.</td><td colspan="3">Solution C. (Boil the solution, which must still smell strongly of H_2S. If there be a yellow precipitate (As_2S_3) add more H_2S and boil, until all the As has been removed; evaporate to a small bulk.) Add a little HNO_3, boil, add NH_4Cl and NH_3 in excess. Filter.</td></tr>
<tr><td rowspan="3">Precipitate D.
Wash, and examine as directed at Table XIII.</td><td colspan="3">Solution D. Add $(NH_4)_2S$, carefully, not in large excess, and heat gently for some time (b). Filter.</td></tr>
<tr><td rowspan="2">Precipitate E.
Wash, and examine as directed at Table XIV.</td><td colspan="2">Solution E (c). Add $(NH_4)_2CO_3$; boil; filter.</td></tr>
<tr><td>Precipitate F.
Wash, and examine as directed at Table XV.</td><td>Solution F (d). Test a small portion with Na_2HPO_4, and stir, to detect Mg (e). Treat the remainder as directed at Table XVI.</td></tr>
</table>

TABLE XI.

Analysis of Precipitate B produced by HCl.

Wash the precipitate off the filter, and boil with a considerable quantity of water. Filter off any undissolved precipitate.

<table>
<tr><td colspan="2">Precipitate B 1. Pour warm NH_3 over it, on the filter.</td><td rowspan="2">Solution B 1. Allow to cool; any crystalline precipitate (of $PbCl_2$) indicates Pb.
Add dil. H_2SO_4.
White precipitate indicates Pb. Confirm by (201).</td></tr>
<tr><td>Precipitate B 2. If grey or black, it is mercurous oxide, and indicates Hg. Confirm by (205).
If white, it is $PbCl_2$ which has escaped solution in the boiling water.</td><td>Solution B 2. Add dil. HNO_3 in slight excess.
White precipitate (AgCl) indicates Ag.
Confirm by (152).</td></tr>
</table>

TABLE XII.

Analysis of Precipitate C produced by H_2S.

Wash until the washings are no longer acid. Pour warm $(NH_4)_2S$ (yellow) over the precipitate upon the filter (*f*).

Precipitate C 1. Treat with successive portions of warm $(NH_4)_2S$, until the liquid gives only a white precipitate (S) with HCl (*g*). Wash, till the washings are no longer precipitated by $AgNO_3$. Pour hot *dil.* HNO_3 over it, warming the acid again and pouring it over the precipitate two or three times (*h*).

Precipitate C 2. Wash till the washings are no longer acid. Pour over it a warm mixture of about 3 vols. HCl and 1 vol. HNO_3.

Ppt. C 3. Wash, dry, and ignite (with the filter if necessary), in a small porcelain crucible. Examine any residue for Sn as in Ppt. C 7.

Sol. C 3. Evaporate to a small bulk (*i*). Add NH_3 in slight excess, and HCl in slight excess. Boil with strips of bright copper. If this be coated, wash it and heat it in a small tube to detect Hg. (218)

Solution C 2. Evaporate to a very small bulk, add *dil.* H_2SO_4, and stir with a glass rod.

Ppt. C 4 indicates Pb.

Sol. C 4. Add NH_3 in excess.

Ppt. C 5. Wash. Dissolve off the filter in very little warm *dil.* HCl. Test a small portion with KI for Bi. (222). Evaporate the remainder to the last drop, and add much water; a milky ppt. indicates Bi.

Sol. C 5. (*Blue* colour indicates Cu). Add HCl till very slightly acid, and much H_2S water. (*Yellow* ppt. Cd.) Collect any black ppt. upon a filter; wash till quite free from acid, boil with *dil.* H_2SO_4, and filter.

Black ppt. CuS.

Solution. Add much H_2S water. *Yellow* ppt. CdS.

Solution C 1. Add *dil.* HCl in slight excess, and a little H_2S. Stir well and filter (*k*). Wash until the washings are no longer acid. Pour warm $(NH_4)_2CO_3$ over it upon the filter (*l*).

Precipitate C 6. Treat, repeatedly, with warm $(NH_4)_2CO_3$ until the liquid no longer gives a yellow ppt. with HCl; wash with water, till the washings are no longer alkaline, and treat the precipitate with a hot mixture of 3 vols. HCl and 1 vol. HNO_3; using as little acid as possible, and pouring it twice or thrice over the precipitate, till nothing but S is left undissolved. Add $(NH_4)_2CO_3$ carefully, in considerable excess, and boil.

Precipitate C 7. Wash. Dry, ignite, with the filter in a porcelain crucible. Fuse the ash with KCy. Stir the cooled fused mass with boiling water. Allow the metallic particles to settle; wash, boil with conc. HCl. Add $HgCl_2$. White or grey ppt. indicates Sn (*m*).

Solution C 7. (*Blue* colour indicates Cu). Add H_2S. Filter off any black ppt. (CuS) and add to the solution *dil.* HCl in slight excess. Orange-red ppt. indicates Sb (*o*).

Solution C 6. Add carefully, *dil.* HCl in excess. Yellow ppt. indicates As. (See notes *m* and *n*.) Confirm (252).

TABLE XIII.

Analysis of Precipitate D produced by NH_4Cl and NH_3.

Wash (preferably with hot water) till the washings are no longer alkaline; dissolve off the filter in as little warm HCl as possible (*p*), evaporate to a very small bulk (*q*), nearly neutralise with $(NH_4)_2CO_3$, add excess of Ammonium Acetate (NH_3 neutralised with acetic acid) and a little Acetic Acid, so that it reddens litmus paper.

<table>
<tr>
<td colspan="4">Precipitate D 1. Wash, dissolve in very little HCl. Add KHO in excess. Boil.</td>
<td colspan="7">Solution D 1. Add at least an equal volume of water, boil for two or three minutes, and filter while hot.</td>
</tr>
<tr>
<td colspan="2" rowspan="2">Precipitate D 2. Dry, ignite, moisten with H_2SO_4. Again dry. Boil with H_2O. Add NH_3 in excess.</td>
<td colspan="2">Solution D 2. Add Acetic Acid in excess.</td>
<td colspan="3">Precipitate D 5. Dissolve in very little hot HCl. Evaporate to a small bulk. Add KHO in excess (*v*). Boil.</td>
<td colspan="4">Solution D 5. Evaporate to a small bulk (filter off any precipitate, and add it to Ppt. D 5); add Fe_2Cl_6, drop by drop, till the liquid is pink (*x*). Add at least an equal volume of water, boil for a minute or two; filter while hot.</td>
</tr>
<tr>
<td>Ppt. D 3. $AlPO_4$ (*s*)</td>
<td>Sol. D 3. Add NH_3 in excess.</td>
<td colspan="2">Precipitate D 6. Dry, ignite, fuse with KNO_3 and Na_2CO_3 (*w*). Treat the fused mass with hot water.</td>
<td rowspan="2">Sol. D 6. Examine in the same manner as Sol. D 2.</td>
<td colspan="2">Precipitate D 7. Dissolve in very little hot HCl. Add NH_3 in excess and $(NH_4)_2S$. Boil and filter.</td>
<td colspan="2">Solution D 7. Evaporate to a small bulk. Add NH_3 in excess and $(NH_4)_2S$. Heat gently.</td>
</tr>
<tr>
<td>Ppt. Fe_2O_3 (brown) (*z*)</td>
<td>Sol. Add Amm. Oxalate Ppt. indicates Ca (*r*)</td>
<td>Ppt. D 4. Al_2O_3 (*t*)</td>
<td>Sol. D 4. Add a mixture of $MgSO_4$ NH_4Cl and NH_3. Stir. Ppt. indicates H_3PO_4.</td>
<td>Residue (Brown) Fe_2O_3 (*z*)</td>
<td>Solution (*Yellow* colour indicates Cr). Add acetic acid in excess and lead acetate; yellow ppt. indicates Cr. (*b b*)</td>
<td>Precipitate contains the Fe which was added to Sol. D 5.</td>
<td>Solution Test for H_3PO_4 as in Sol. D 4. (*y*)</td>
<td>Ppt. D 8 may contain ZnS *white*, MnS *buff*, CoS *black*, NiS *black*. Examine as directed for Ppt. E. Table XIV.</td>
<td>Sol. D 8 may contain Ca, Ba, Sr, and Mg, which existed originally as phosphates. Examine as directed for Solution E, Table X. (*a a*)</td>
</tr>
</table>

TABLE XIV.

ANALYSIS OF PRECIPITATE E PRODUCED BY $(NH_4)_2S$ (*cc*).

Wash (preferably with hot water) till the washings are colourless, pour hot *dil.* HCl over it, upon the filter (any black residue is probably CoS or NiS; dissolve it in a little warm HNO_3, and add it to the HCl solution). Evaporate to a very small bulk, and add KHO in excess.

<table>
<tr>
<td colspan="3">PRECIPITATE E 1 (ee). Dissolve off the filter in very little hot HCl (brown colour indicates Mn). Evaporate to a very small bulk, blue colour indicates Co. Add $(NH_4)_2CO_3$ in excess and boil.</td>
<td rowspan="3">SOLUTION E 1. Add $(NH_4)_2S$. Precipitate probably indicates Zn. Confirm (dd).</td>
</tr>
<tr>
<td rowspan="2">PRECIPITATE E 2 (ff). Wash, dry, ignite, and fuse with KNO_3 and Na_2CO_3. Green or blue mass indicates Mn.</td>
<td colspan="2">SOLUTION E 2. Add KCy in slight excess; boil; add HCl in excess. Boil and add HCl till there is no more smell of HCy. Add KHO in excess, and boil till there is no more smell of NH_3.</td>
</tr>
<tr>
<td>PRECIPITATE E 3. Wash, and test for Ni with a bead of borax in the blowpipe flame (142).</td>
<td>SOLUTION E 3. Add HNO_3 in excess. Evaporate to dryness, and fuse the residue. Stir the fused mass with boiling water. Collect the undissolved residue on a filter, and test it for Co with a bead of borax in the blowpipe flame (127).</td>
</tr>
</table>

TABLE XV.

ANALYSIS OF PRECIPITATE F PRODUCED BY $(NH_4)_2CO_3$.

Wash till the washings are no longer alkaline. Dissolve off the filter in very little *dil.* HCl, and evaporate to dryness. (Test a little of the residue on a moist Pt wire, in the blowpipe flame (169)). Dissolve in a small quantity of water; divide into two parts.

<table>
<tr>
<td rowspan="4">PART 1.
To a small portion add $CaSO_4$. Immediate ppt. indicates Ba. See Part 2.

Ppt. only on standing or warming indicates Sr. See Part 2, beginning at "Add dil. H_2SO_4."</td>
<td colspan="4">PART 2.
Add AMMONIUM ACETATE (prepared by adding acetic acid to ammonia till it is slightly acid) and POTASSIUM BICHROMATE till an orange colour appears.</td>
</tr>
<tr>
<td rowspan="3">YELLOW PPT.
$BaCrO_4$. Wash and test by coloured flame test (169).</td>
<td colspan="3">SOLUTION. Divide into two parts.</td>
</tr>
<tr>
<td rowspan="2">To a small part add $CaSO_4$ and heat. Ppt. indicates Sr.</td>
<td colspan="2">To the remainder, add dil. H_2SO_4. Stir, and set aside for a few minutes.</td>
</tr>
<tr>
<td>PPT.
$SrSO_4$ and perhaps some $CaSO_4$.</td>
<td>SOLUTION.
Add NH_3 in excess and AMMONIUM OXALATE. Precipitate indicates Ca.</td>
</tr>
</table>

See *gg* for another method of separating Ba, Sr, and Ca.

TABLE XVI.

Analysis of Solution F* filtered from the Precipitate by $(NH_4)_2CO_3$.

Evaporate to dryness in a small porcelain or platinum dish, and heat the residue until no more white fumes (NH_4Cl) are given off. If *no* Mg has been detected, *omit* all the following parenthesis. (Dissolve the residue in a little water, add BARYTA-WATER till it is strongly alkaline, boil; filter from the precipitated magnesium hydrate, add $(NH_4)_2CO_3$, boil and filter from the barium carbonate. Evaporate the filtered liquid to dryness, and heat the residue until no more white fumes are given off.) Test the residue on Pt wire by the coloured flame test (169). Dissolve in *very little* water, filter if necessary, and divide into three parts (*hh*).

Part 1.	Part 2.	Part 3.
Add two or three drops of a strong solution of Tartaric Acid from the end of a glass rod. Stir briskly, and set aside for half an hour (356).	Add two or three drops of a strong solution of $PtCl_4$. Stir, and set aside for half an hour (354).	Add a drop of KHO (to render the liquid alkaline if it be not so) and two or three drops of a strong solution of POTASSIUM ANTIMONIATE. Stir briskly, and set aside for half an hour (358).

Ammonium must be tested for by boiling the original substance with KHO in excess (360).

* Evidently, if no metal (or only Mg) has been found in the preceding portion of the analysis, the original substance may be examined here instead of evaporating Solution F. It must be tested for ammonium (360), and, should this be found, the substance must be ignited until all ammoniacal salt is expelled, and further treated as above. If NH_4 be absent, the ignition may be omitted.

EXAMINATION OF COMPLEX SUBSTANCES FOR NON-METALS AND ACIDS.

The analyst has a portion of the solid substance at his disposal. If not, *see* Tables XVII., XIX.

519 HEAT A SMALL PORTION OF THE SOLID UPON A PIECE OF BROKEN PORCELAIN, OR A SCRAP OF PLATINUM FOIL.

(*a*) **Blackening, with evolution of a peculiar odour;** *Tartaric, Citric, Gallic, Tannic, Benzoic* (odour of frankincense), *Citric, Acetic* or *Hydroferrocyanic* (odour of ammonia) acid may be present.

(*b*) **Sulphurous odour;** *Sulphur* is present, either as a *Sulphide*, a *Hyposulphite*, or, less probably, a *Sulphite* or *Sulphate*.

(*c*) **Rapid combustion** or **explosion;** a *Nitrate* or *Chlorate*, together with carbon or some organic substance, or sulphur, or phosphorus (white flame and thick smoke).

520 HEAT A LITTLE OF THE SOLID IN A GLASS TUBE (478). Some of the above phenomena may be observed, and, in addition,

(*a*) **Deposition of moisture,** which may be due to the presence of *Water* in the solid, or to its production from some organic substance.

(*b*) **Brown nitrous fumes;** indicating a *Nitrate*.

(*c*) **Violet fumes;** *Iodine* or an *Iodide*.

(*d*) **Cyanogen is evolved** (burning with a pink flame); a *cyanide* or *ferrocyanide*.

521 HEAT A SMALL PORTION OF THE SOLID WITH STRONG SULPHURIC ACID.

Refer to (479) for the phenomena likely to be observed. In dealing with a complex substance, it must be remembered that

Carbonic acid may either be derived from a carbonate, or from the reaction between an oxidizing agent (such as chromic acid or binoxide of manganese) and organic matter.

Chlorine may be derived from a hypochlorite, or from the reaction between a chloride and an oxidizing agent

(such as nitric acid, chromic acid, or binoxide of manganese).

522 HEAT A LITTLE OF THE SOLID WITH DILUTE HYDROCHLORIC ACID.

Refer to (480) for the phenomena likely to be observed. In addition to the remarks made in (521), it must be here borne in mind that

Hydrosulphuric acid may be derived from a sulphide, or from the reaction between a sulphite or hyposulphite and some deoxidizing agent (such as metallic zinc), in the presence of the hydrochloric acid.

523 Boil twenty or thirty grains of the powdered substance with water, filter the solution, if necessary, and reserve the undissolved portion for examination by Table XX. Examine the solution as in (524).

524* To a portion of the solution, add DILUTE SULPHURIC ACID, and heat gradually to boiling.

(*a*) **Effervescence** takes place even in the cold, and a nearly inodorous gas is evolved; presence of *Carbonic Acid.* Confirm by (422).

(*b*) The **odour of burning sulphur** is perceptible; presence of *Sulphurous* or *Hyposulphurous Acid* (in the latter case, sulphur will separate).

(*c*) The gas has an **odour of putrid eggs;** presence of *Hydrosulphuric Acid* or of *Sulphur* combined with a metal.

(*d*) The **smell of chlorine** is perceived; presence of *Hypochlorous* or of *Chloric Acid.* Confirm by (394, 395*a*, 397).

(*e*) **Hydrocyanic Acid is smelt** at the mouth of the tube; presence of *Hydrocyanic Acid*, or a *Cyanide*, or of *Hydroferrocyanic Acid*, or a *Ferrocyanide.* Confirm by (436, 440).

(*f*) *Acetic and Benzoic Acids* will be recognised by their characteristic odours.

Proceed with the examination of the solution according to Table XVII. or XIX.

* It is of course impossible that this solution can contain *all* the bases and acids in the list, since several of them would form salts which are insoluble in water, as baryta and sulphuric acid, oxide of silver and hydrochloric acid, &c. It is consequently unnecessary, after having determined the bases in a mixture of salts soluble in water, to look for any acids which form with them salts that are insoluble. (See Table of Solubilities in the Appendix.)

TABLE XVII.

ANALYSIS OF A COMPLEX MIXTURE FOR THE ACIDS AND NON-METALLIC BODIES.

CASE 1.—*The substance has been dissolved in water, and the solution is not strongly acid.*

(This may be inferred if it does not effervesce on adding a little sodium carbonate.)

PART 1.	PART 2.	PART 3.		PART 4.	PART 5.	PART 6.
Add $Ba(NO_3)_2$ *(ii)* Precipitate Add *dil.* HNO_3 *Undissolved* $BaSO_4$ indicates a *sulphate.* *Dissolved* Barium *phosphate, borate, oxalate, sulphite, fluoride, chromate* (yellow) *tartrate, citrate,* [hyposulphite, carbonate, arsenite, arseniate, silicate], *(kk).*	Add $AgNO_3$ Precipitate white Silver *chloride, cyanide, sulphocyanide, ferrocyanide, borate, oxalate, sulphite, tartrate, citrate, benzoate* [carbonate, silicate, hyposulphite (376*a*)]. *Yellow* *phosphate, iodide* [arsenite] *Brown (ll)* *ferricyanide* [arseniate] *Red* *Chromate* *Black* [Sulphide]. Examine according to Table XVIII.	Add $CaCl_2$ *(mm).* Precipitate. Add ACETIC ACID.		Add Fe_2Cl_6, drop by drop *(nn)* Precipitate Ferric *phosphate* (white) [Arseniate (white)] [Sulphide (black)] *(oo)* *ferrocyanide* (blue), *gallate* (blue-black), *tannate* (blue-black), *benzoate* (buff), *(pp).* Colour Ferric *sulphocyanide* (dark red) (434), *acetate* (light red) (432), *ferricyanide* (dark brown) (441), *gallate* or *tannate* (inky blue), *sulphite* or *hyposulphite* (transient red or purple).	Test for a *nitrate* by adding $FeSO_4$ and, *very carefully,* CONC. H_2SO_4 Brown band (366, 367).	The following acids may still have escaped detection, and should be specially tested for: *Chloric* (394), *Hypochlorous* (397), *Hydrocyanic* (436), *Boracic* (411, 412), *Tartaric* (444), *Citric* (451), *Acetic* (432).
		Undissolved Calcium *oxalate* (427, 430), *fluoride* (405).	*Dissolved* Calcium *phosphate, borate, sulphite, tartrate.* [Carbonate, arsenite, arseniate, silicate]. Add a drop of Fe_2Cl_6 from a glass rod. White ppt. Ferric *phosphate* [or arseniate]. (382, 383).			

TABLE XVIII.

ANALYSIS OF A COMPLEX MIXTURE FOR THE ACIDS AND NON-METALLIC BODIES. EXAMINATION OF THE PRECIPITATE PRODUCED BY $AgNO_3$.

Wash the precipitate, and treat with *dil.* HNO_3.

<table>
<tr><td colspan="6">UNDISSOLVED.
Silver chloride, cyanide, sulphocyanide, ferrocyanide, ferricyanide (brown), iodide (yellow), [sulphide, black]. Filter off, wash, and pour NH_3 over it.</td><td rowspan="4">DISSOLVED.
Silver phosphate (yellow), chromate, borate (416), oxalate, sulphite (374), tartrate, citrate, benzoate [arsenite, arseniate, carbonate, silicate, hyposulphite].</td></tr>
<tr><td colspan="3">UNDISSOLVED.
Silver iodide, ferrocyanide, [sulphide (black)]. Wash, and boil with HNO_3.</td><td colspan="3">DISSOLVED.
Silver chloride, cyanide, sulphocyanide, ferricyanide. Evaporate to dryness, ignite the residue. Heat with NH_3.</td></tr>
<tr><td colspan="2">UNDISSOLVED.
Silver iodide, ferricyanide (brown), (formed from the ferrocyanide). Treat with NH_3.</td><td rowspan="2">DISSOLVED.
Silver sulphide.</td><td colspan="2">UNDISSOLVED.
Metallic silver, from the other three silver-salts. Ferric oxide from the ferricyanide. Boil with dil. HNO_3.</td><td rowspan="2">DISSOLVED.
Silver chloride; add HNO_3 in excess. White ppt. indicates a chloride.</td></tr>
<tr><td>UNDISSOLVED.
Silver iodide (399).</td><td>DISSOLVED.
Silver ferricyanide (formed from the ferrocyanide).</td><td>UNDISSOLVED.
Brown Fe_2O_3 indicating a ferricyanide.</td><td>DISSOLVED.
Silver; test with HCl.</td></tr>
</table>

TABLE XIX.

ANALYSIS OF A COMPLEX MIXTURE FOR THE ACIDS AND NON-METALLIC BODIES.

CASE 2.—*The solution of the substance in water is strongly acid.*

Add Na_2CO_3 till the solution is strongly alkaline, boil for a minute or two, filter from any precipitate (containing the metals), acidulate slightly with HNO_3, heat gently to expel CO_2.

PART 1.	PART 2.	PART 3.		PART 4.
Add $BaCl_2$ Precipitate indicates a *sulphate.*	Add $AgNO_3$ Precipitate. Examine by the method adopted in Case 1, for that portion of the ppt. by $AgNO_3$ insoluble in HNO_3. See Table XVIII.	Add NH_3 till the solution is alkaline and divide into two parts.		Test specially for *sulphurous* (372), *citric* (451), *acetic* (432), *boracic* (411, 412), *chloric* (394, *et seq.*) *acids.*
		To one portion add $CaCl_2$ Precipitate. Examine by the method adopted for Part 3 in Case 1.	Evaporate the other portion gently until it is neutral, and add Fe_2Cl_6, drop by drop. Refer to the examination of Part 4 in Case 1.	

TABLE XX.

Analysis of a Complex Mixture for the Acids and Non-metallic Bodies.

Case 3.—*The substance is insoluble in water, but dissolves in acids.*

1.	2.	3.						4.
Boil a portion of the substance with HCl, filter, if necessary, and test for *sulphates* with $BaCl_2$ (370).	Boil a portion of the substance with HNO_3, filter, if necessary, and test for *chlorides* with $AgNO_3$ (391).	Boil a portion of the substance with Na_2CO_3 and a little water for a minute or two; filter, and divide into six parts.						Examine the original substance for a *sulphide* (378, 380), or a *carbonate* (422).
		Part 1. Examine as in Case 2 Table XIX. (beginning at "acidulate slightly.")	Part 2. Test for *nitrates* by adding *dil.* H_2SO_4 in slight excess; and $FeSO_4$ and, *very carefully*, conc. H_2SO_4 (367).	Part 3. Add excess of HCl and observe any indication of a *sulphite* (371, 372), a *sulphide* (378), or *hyposulphite* (375).	Part 4. Add Acetic acid in excess, and Fe_2Cl_6. Blue precipitate indicates a *ferrocyanide*. Dark brown colour indicates a *ferricyanide*. White precipitate indicates a *phosphate* (388).	Part 5. Add $FeSO_4$, Fe_2Cl_6, and excess of HCl. Blue precipitate indicates a *cyanide*.	Part 6. Neutralise exactly with HNO_3, and add Fe_2Cl_6. Dark red colour indicates a *sulphocyanide*.	

Notes to Table X.

a. When much nitric acid has been used in dissolving the substance, the solution should be evaporated to a very small bulk, and diluted with water acidified with hydrochloric acid, before testing with hydrosulphuric acid.

b. Small quantities of manganese may not give any precipitate unless the liquid be allowed to stand for twelve hours.

c. If this filtered liquid is brown, it probably contains a little sulphide of nickel, or possibly of cobalt, and must be evaporated until the excess of sulphide of ammonium is expelled (known by the odour), slightly acidulated with dilute hydrochloric acid, the precipitate collected upon a filter and tested by the blowpipe (142), and the filtered liquid mixed with ammonia and carbonate of ammonia. In some cases, when much organic matter is present, the filtrate from the hydrosulphate of ammonia precipitate may still contain iron, chromium, &c. In such a case it must be evaporated to dryness, calcined till the organic matter is consumed, the residue dissolved by digesting in hydrochloric acid, and the examination recommenced from Solution C.

d. If this solution be very dilute, it should be concentrated by evaporation. It is also advisable to test it with a little oxalate of ammonia, to be quite sure that the baryta, strontia, and lime have been entirely removed.

e. A slight flocculent precipitate of aluminium phosphate is occasionally obtained here.

Notes to Table XII.

f. If the precipitate be very large, it may be rinsed from the filter into a small beaker, and heated with ammonium sulphide.

g. When copper is present, this point is difficult to attain, and two or three washings with ammonium sulphide must suffice.

h. If the precipitate be very large, rinse it into a beaker, and boil it with the acid.

i. When gold is present in the substance analysed, it is

often found here, and may be detected by adding excess of stannous chloride or of ferrous sulphate, when a purple brown precipitate is obtained.

k. If the precipitate be pure white, it is only sulphur, and need not be examined.

l. If the precipitate be large, rinse it into a beaker, and heat it gently with ammonium carbonate. A solution of ammonium sesquicarbonate saturated in the cold, is best adapted for this purpose.

m. When bisulphide of tin is mixed with sulphide of arsenic, it is also liable to be dissolved by ammonium sesquicarbonate. In case of doubt, the precipitate produced by hydrochloric acid in the carbonate of ammonia solution should be washed till free from that acid, dried, and added, together with the filter in small pieces, to some nitrate of potash fused in a porcelain crucible. The cool fused mass is treated with boiling water (which will generally leave a residue, if tin is present), enough nitric acid added to render it slightly acid, and boiled. Any precipitate is collected upon a filter and examined for tin by incinerating the filter containing it, fusing the ashes with cyanide of potassium, washing any reduced metal with water, boiling with hydrochloric acid, and testing with mercuric chloride for tin. The solution filtered from the precipitate produced by nitric acid is mixed with an excess of ammonia, and a mixture of sulphate of magnesia, chloride of ammonium, and ammonia added; on stirring, a crystalline precipitate of arseniate of magnesia and ammonia will indicate the presence of arsenic.

n. Ascertain whether the arsenic existed as arsenious or arsenic acid by special tests applied to the original solution.

o. Special tests must be applied to the original solution to ascertain the state of the tin (whether as a stannous or stannic compound), and of the antimony (whether as antimony oxide or antimonic acid).

Notes to Table XIII.

p. Any white gelatinous residue which refuses to dissolve is probably silica, and should be tested by the blowpipe.

q. If the presence of silica be suspected, evaporate to

dryness, and redissolve in a little warm dil. HCl, which will leave the silica undissolved.

If there is reason to believe, from the green or purple colour of this solution, that it contains much chromium, add sodium carbonate till it is strongly alkaline, boil for some minutes and filter.

Test the filtered solution for phosphoric acid with a mixture of magnesium sulphate, ammonium chloride, and ammonia.

Dry the precipitate (burn the filter, if necessary), and fuse it with about one part of sodium carbonate and three parts of potassium nitrate, on platinum foil, or in a porcelain crucible.

On cooling, stir the fused mass with boiling water, and filter. (Green solution indicates manganese; yellow solution, chromium.) Acidulate the solution with acetic acid and boil. Filter off any brown precipitate (MnO_2), and divide the solution into two parts; test one for chromium (CrO_3) with lead acetate; mix the other with ammonia in excess; filter off any precipitate (Al_2O_3), and test the solution for phosphoric acid with a mixture of magnesium sulphate, ammonium chloride, and ammonia.

The residue left undissolved by boiling water is washed, dissolved in hot hydrochloric acid, and mixed with ammonia in excess. Filter off the precipitate and examine by Table XIII.

r. This calcium was most probably present in the original substance in the form of oxalate or fluoride of calcium. Examine the original substance for oxalic and hydrofluoric acid by (430, 406).

s. This precipitate should be dissolved in nitric acid and tested for phosphoric acid with ammonium molybdate.

t. This alumina must have existed in Precipitate 1, in the form of aluminium phosphate. A small quantity of alumina is often derived from the potash.

u. A white precipitate here is probably alumina, a brown precipitate, ferric oxide, and a green precipitate, chromic oxide.

v. A green colour of this solution indicates chromium. The ordinary potash of the laboratory is liable to contain alumina and phosphoric acid. Pure potash or soda should

be employed when small quantities of these bodies are sought after.

w. A yellow mass indicates chromium, a green, manganese.

x. A precipitate indicates phosphoric acid.

y. This phosphoric acid was probably contained in Precipitate D as a phosphate of calcium, barium, strontium, or magnesium.

z. Examine the original solution to ascertain whether the iron existed there as a ferrous or a ferric compound (*see* note *l* on page 144).

aa. When much iron or aluminium is present, magnesium and calcium are often carried down in the precipitate produced by ammonia, although no phosphoric acid existed in the solution.

bb. The state in which the chromium existed in the original solution must be ascertained by special tests.

Notes to Table XIV.

cc. A white precipitate is probably due to zinc; a buff precipitate to manganese; a black precipitate to cobalt or nickel.

dd. This precipitate is often dark-coloured, from the presence of lead derived from the potash, or of iron which has escaped precipitation in consequence of the presence of organic matter. To confirm the presence of zinc, collect the precipitate on a filter, wash it, dissolve in very little nitric acid, add ammonia in excess, filter off any precipitate (of oxide of lead or iron), and add potassium ferrocyanide; a white precipitate indicates zinc. If the quantity be sufficient, another portion of the nitric solution may be mixed with excess of sodium carbonate, boiled, the precipitate collected on a filter, dried, and tested for zinc by the cobalt nitrate test (163).

ee. A blue precipitate indicates cobalt; a green, nickel; a brown, manganese.

ff. Test this precipitate (E 2) with a borax bead for cobalt, which may be carried down with the manganese carbonate.

NOTE TO TABLE XV.

gg. Another method of analysing the precipitate produced by ammonium carbonate is the following:—Dissolve in hydrochloric acid, evaporate to dryness. Treat the residue with alcohol (*absolute* if obtainable): barium chloride is left undissolved. Mix the filtered solution with water, and add hydrofluosilicic acid to precipitate any dissolved barium; filter the solution and test for strontium and calcium as in Table XV., omitting the addition of ammonium acetate and potassium bichromate.

NOTE TO TABLE XVI.

hh. The detection of potassium and sodium in complex mixtures requires very careful work. The residue should be dissolved in about 10 or 12 drops only of water, and the solution dropped from the filter into three watch-glasses.

NOTES TO TABLE XVII.

ii. $BaCl_2$ may be employed, unless lead, silver, or (mercurous) mercury is present.

kk. Substances enclosed in brackets would have been detected in an earlier stage of the analysis.

ll. If the original solution contained a free alkali, a brown precipitate of Ag_2O may be obtained here.

mm. If the original solution contained lead, silver, or (mercurous) mercury, these must be precipitated by sodium carbonate before adding calcium chloride.

nn. If the solution contains lead, silver, or (mercurous) mercury, it must be boiled with sodium carbonate, filtered, and carefully neutralised with nitric acid before applying this test.

oo. If an iodide be present, a black precipitate of iodine may be formed here.

pp. If the original solution be alkaline, a brown precipitate of ferric hydrate may be obtained here.

PART IV.

QUANTITATIVE ANALYSIS.

Introductory Remarks.

525 In the processes which I have now described, the object of the experimenter has been to ascertain what substances are present in a given salt or mixture of salts, which branch of analysis is called *qualitative*. I will now detail a few processes which have for their object the determination of the quantity of the ingredients of saline compounds: this branch of analysis is called *quantitative*. It is not my intention to enumerate the methods which have been devised for the separation and estimation of all, even of the more common compounds, but merely to give the student a general idea of the subject, by conducting him through a few simple examples of quantitative analysis, referring him, if he wishes for more extended information, to the larger works of Rose and Fresenius.*

I will first briefly describe some of the more important operations which have to be performed in the course of a quantitative analysis; and the student must bear in mind that the more care he bestows upon them, the more correct will be his results; as the loss of a single drop of liquid, or the presence of a very small quantity of soluble matter left in a precipitate, owing to carelessness in washing, will often occasion serious errors.

* Traité Pratique d'Analyse Chimique, par H. Rose, of which an English translation by Dr. Normandy has been published.
Chemical Analysis, Qualitative and Quantitative, by C. R. Fresenius, translated by Vacher.

CHAPTER I.

OPERATIONS IN QUANTITATIVE ANALYSIS.

Pulverization.

526 Most substances may be reduced to sufficiently fine powder for analysis, by pounding in a common Wedgwood mortar; in some cases, however, it is first necessary to break the substance into small fragments, in one of iron or gun-metal; or in default of this, the substance may be loosely wrapped in strong brown paper, and struck with a hammer. When the substance is difficult of solution, as in the case of some silicious minerals, it is sometimes necessary to reduce it to an impalpable powder in a small agate mortar; and on the fineness of this pulverization the success of an analysis often depends.

Drying.

527 Many substances, especially when in the state of powder, absorb moisture from the atmosphere, which, of course, adds to their weight. Before weighing out accurately the quantity of the substance for analysis, it is therefore necessary to deprive it of this hygroscopic moisture. This is generally done by heating it in a small basin on the water-bath or sand-bath, care being taken that the heat does not rise so high as to cause decomposition. The hot-water oven shown in figure 67, is very convenient for drying substances at a low temperature; the sides are made hollow, and filled with water, so that the temperature inside never rises higher than 212° F. (100° C.). When a substance thus exposed ceases to lose weight, on being weighed at short intervals, it may be considered suffi-

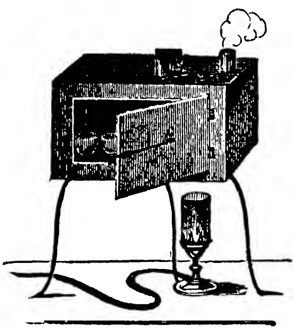


Fig. 67. Hot-Water Drying Oven.

ciently dry. By using saline solutions, which boil at a higher temperature than water, a steady heat, considerably higher, may be obtained.*

Weighing.

528 Either 20, 25, or 33·3 grains will generally be found the most convenient quantity to take for quantitative experiments, regard being had to the number of constituents to be estimated, and the quantity of the substance at our disposal. The quantity may depend also on the method we intend to pursue, whether we propose to estimate all the ingredients from the same portion, or from two or more separate portions of the substance. If 20 grains are used, the results multiplied by five will give the percentage; or if 25 or 33·3, they must be multiplied by four or three. For most purposes the student will find a balance which is capable of weighing within one-tenth of a grain sufficiently accurate; and it should be furnished with weights from one-tenth of a grain to 1000 grains.

529 A substance should never be weighed while warm, as it causes an upward current of air in its vicinity, which tends to buoy it up, and makes it appear to weigh lighter than it really is. In quantitative analysis, it is, of course, necessary to avoid the slightest loss in the weighed portion, as a deficiency in the weight of the ingredients would be the consequence, and the accuracy of the analysis would be seriously interfered with. Most substances in the state of fine powder, especially after having been recently ignited, are very prone to absorb moisture from the air; to obviate this, which would add materially to their weight, such substances should be weighed in a covered crucible, as soon as possible after cooling.†

530 When, as is frequently the case, especially with liquids, a substance has to be weighed in a flask, dish, or other vessel, the latter may either be counterpoised with strips of lead or shot, which are conveniently placed in a pill-box; or its weight may be previously noted, and afterwards deducted from the gross weight.

* Taylor's air-bath is now very generally used for drying substances.

† The cooling should be allowed to take place under a glass shade, in which a dish of strong sulphuric acid is kept, in order to dry the air.

531 If the substance is weighed in a watch-glass, the last particles should be brushed off with a camel's hair pencil into the vessel to which the substance is transferred.

Solution.

532 Before the ingredients of a substance can be determined, either qualitatively or quantitatively, it is necessary to bring the substance into solution. For this purpose water is to be preferred when the substance dissolves readily in it; and in the case of those compounds which are insoluble in water, one of the acids (generally hydrochloric) is employed, which has been found in the course of the preliminary examination to be the best adapted for the purpose.

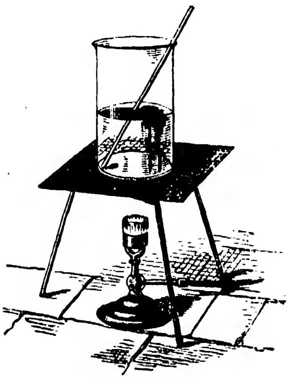


Fig. 68.

The dissolution of a substance is almost invariably assisted by heat, so that it is always advisable to use a vessel for the purpose which can be heated over a lamp without danger of fracture, as a small beaker or glass flask. (Figs. 68 and 69.) The latter has the advantage

Fig. 69.

Fig. 70.

of preventing loss by ebullition or spurting, as any particles of liquid that may be projected from the surface during ebullition, fall against the inner surface, and run back into the flask, especially if it is placed in an inclined position over the lamp. Occasional stirring facilitates the solution, and, as a general rule, the more finely the substance has been pounded, the more readily it dissolves. When a substance has to be digested in acid for a length of time, with the aid of heat, the evaporation of the acid may be in a great measure prevented, by placing a small glass funnel in the mouth of the flask (fig. 70); the acid condenses, and runs back into the flask.

Precipitation.

533 When a substance is obtained in solution, the various compounds present are in most cases separated for the purpose of estimation, by adding to it some solution, which causes one or more of the ingredients to precipitate in the solid state; as, when we wish to estimate the quantity of sulphuric acid in any solution, we add to it a solution of chloride of barium, which, if added in sufficient quantity, causes the whole of the acid to precipitate in the form of sulphate of baryta, which being insoluble in water, may be washed without loss, and when dry is weighed; the weight of the sulphuric acid which it contains may then be calculated from it.

Precipitation is usually effected in beaker glasses of the form shown in fig. 68. When precipitating a substance in quantitative analysis, it is important that sufficient of the precipitant be added to throw down *the whole* of the substance affected by it, as otherwise a deficiency in weight would be occasioned: this is easily ascertained by adding a drop of the precipitant to the solution filtered from the precipitate, which will cause a further precipitate if sufficient had not before been added. When the precipitate is at all soluble, as the bitartrate of potash, or ammonio-phosphate of magnesia, it is always advisable to allow the mixture to stand several hours before filtering, in order to insure the separation of the whole of the required salt. When the whole of the precipitate is thrown down, it is separated from the solution either by filtration or decantation.

Filtration.

534 The process of filtration is that most commonly adopted for separating a precipitate from the solution in which it was formed. The paper best adapted for the purpose is a thin white blotting-paper, which should be free from visible holes, and should leave, when burnt, only a minute trace of inorganic matter.* Such a paper may be purchased from any of the dealers in chemical apparatus. It is convenient to keep a stock of filters ready cut, of a circular form, and of sizes varying from three to ten inches diameter. These may be made by having circular pieces of tin plate of the different sizes, and scoring round them with a pencil upon the paper, when several sheets may be cut through at once with scissors.

535 The filter when required for use, is folded twice at right angles (fig. 71), opened out into a conical form, and placed in a glass funnel (fig. 72), the sloping sides of which should open at an angle of about 60°, and should merge into the neck at a definite angle, when it will be found to match the form of the folded filter, and will support it uniformly throughout. When placed in the funnel the paper is moistened with water, for the purpose of causing the fibres to expand, and thus diminishing the sizes of the pores, without at the same time choking them with solid particles: if this is not done, and a solution mixed with a precipitate is poured into the dry filter, some of the finely divided particles of the precipitate are drawn into the pores by capillary attraction, and tend to prevent the passage of the clear solution through them. The filter should never be allowed to reach higher than the top of the funnel, as otherwise the weight of the liquid might cause the paper to give way; and there would also be danger of some of the solution running down the outside of the funnel, after passing through the projecting paper. It is even better that the funnel should project half an

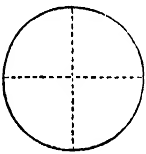


Fig. 71.

Fig. 72.

* For many precipitates (sulphate of baryta, *e.g.*), the Swedish filtering paper is to be preferred to all other kinds.

inch above the edge of the filter. When the filter is thus prepared, it may be supported either on the ring of a retort stand (fig. 73) or on a perforated block of wood placed on the glass intended to catch the filtered solution, the hole being made to fit the funnel.

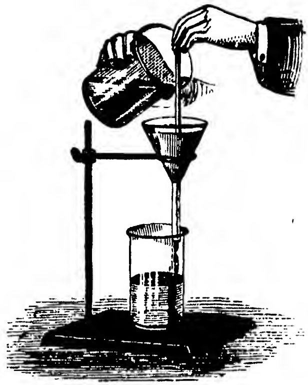


Fig. 73.

536 The solution to be filtered should be poured gently down a glass rod (fig. 73), so as to fall on one of the slanting sides of the filter, and not into the apex, as that would endanger the bursting of the paper, and cause splashing. A liquid, holding a precipitate in suspension, should never be poured higher than within half an inch of the margin of the filter. When the whole of the mixture has been poured on the filter,* fresh water should not be added for the purpose of washing, until the whole of the solution has passed through; then, by means of a washing-

Fig. 74.

* In removing the last particles of the precipitate from the vessel in which it has been deposited, a glass rod, with one end covered with a cap of caoutchouc, will be found very convenient; in the case of a flask, the rod may be bent, or a tuft at the end of a quill may be used.

bottle (101), the precipitate left on the filter is well washed; the current of water being applied first towards the upper part of the filter, and directed gradually downwards (fig. 74). When the filter has been thus nearly filled up with water, allow the whole to run through before adding any more, and then repeat the washing, until a drop of the filtered liquid leaves no fixed residue when evaporated on a piece of glass. If the precipitate, while standing in the filter, cakes together into lumps, these must be broken up by directing upon them a strong current of water from the washing-bottle, as otherwise the water would not penetrate them, and some of the soluble matter would escape removal.*

Fig. 75. Hot Water Filtering Stand.

537 It is sometimes necessary to keep the mixture hot during filtration, to prevent any of the soluble ingredients solidifying: this may be done very conveniently, by placing the funnel in a zinc or copper box of the form shown in fig. 75, which may be kept full of hot water, and boiling, if necessary, over a lamp.

538 The liquid is generally filtered into a beaker glass, and occasionally into flasks or dishes; it is always advisable to cause the stream to run gently down the side of the vessel, and not to fall drop by drop into the centre of the glass, as this would cause splashing and probably some loss. It occasionally happens that some of the precipitate passes through with the filtered solution, as may be seen in the case of freshly precipitated oxalate of lime or sulphate of baryta. When this takes place, it is sometimes

* Many precipitates are washed with hot water; for this purpose a flask must be employed which is furnished with tubes like the washing-bottle, and can be heated over a lamp. In many cases, for example, in that of oxide of copper precipitated by potash, it will be found advantageous to allow the precipitate to subside, decanting the clear liquid by degrees through the filter, and to boil the precipitate with water, afterwards allowing it to subside as before. When this process has been repeated once or twice, the precipitate may be thrown upon the filter, and the washing completed.

necessary to pass it through the filter twice or three times before it comes through quite clear. This may, however, in most cases, be obviated by boiling the mixture before filtering, which causes the finely divided particles of the precipitate to aggregate together. The presence of some saline matters in solution, also, sometimes prevents a precipitate passing through: chloride of ammonium, for example, exerts this property with sulphate of baryta.

539 When the precipitate on the filter is completely washed, the funnel, with its contents, is placed on a small tripod, or retort stand, on the warm sand-bath, or near a fire, when the precipitate will gradually dry; it may then be separated from the filter, ignited in a small platinum or porcelain crucible (unless decomposable at a high temperature), and weighed.

540 Precipitates may be very rapidly dried by supporting the funnel and filter containing them in a beaker with the bottom out, and placing this upon a piece of strong coarse wire network, resting upon a tripod stand (fig. 76), over an Argand burner with a very small flame. In order to prevent any dust from falling into the precipitate, it is advisable to cover the funnel with a piece of filter-paper, secured by folding down over the rim.

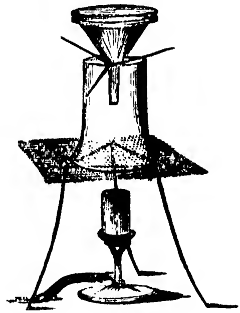


Fig. 76.

541 In cases where the quantity of the precipitate is very small, and where it will not bear a red heat without decomposition, it may be weighed in the filter, which should be placed in a covered porcelain crucible of known weight, dried at 212°, and weighed before the precipitate is collected upon it. When the precipitate on the filter has been thoroughly washed, the latter is partly dried in the funnel, placed in the crucible, dried as before, and as soon as it is cold, again weighed; when the increase in weight will, of course, be that of the precipitate. A light beaker, about $2\frac{1}{2}$ inches by $1\frac{1}{2}$ inch, covered with a light watch-glass, is very convenient for

weighing dry filters. Their weights should be now written upon them with a diamond.

542 It is often necessary, before weighing a precipitate, to burn the filter containing it. After the greater part of the precipitate has been removed by gently rubbing the sides of the filter-paper against each other, the filter is held with a pair of pliers or in a piece of platinum wire, and set fire to, over the crucible in which the precipitate is to be ignited, the crucible being placed in a basin, or upon a sheet of glazed paper, in case any of the ashes should fall over its sides (fig. 77); these are then collected and ignited in the open crucible, together with the portion of the precipitate previously removed from the filter, until the whole of the charcoal derived from the paper is burnt away. In cases of great accuracy, the weight of the paper ashes, ascertained by weighing those derived from six or eight similar filters, must be deducted from the gross weight; when the paper is good, however, it does not contain more than one to three-thousandths of its weight of inorganic matter,* so that this precaution is scarcely necessary in ordinary cases of analysis.

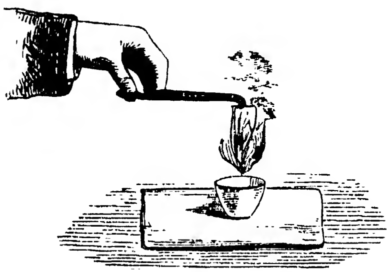


Fig. 77.

Decantation.

When a precipitate is found to subside rapidly to the bottom of the liquid, and when it is known to be very insoluble in water, it may be washed by DECANTATION, instead of on a filter, and in many cases this is the more expeditious method. The mixture is placed in an upright jar or beaker, which is then filled up with water, and allowed to stand until the precipitate has subsided

* Consisting chiefly of silica and alumina, but containing a little lime, magnesia, and peroxide of iron, with carbonic acid and sulphuric acid.

to the bottom, leaving the supernatant liquid clear. The latter is then removed with a syphon (fig. 78), or carefully poured off, and the jar again filled up with distilled water, the process being repeated until all the soluble matter has been removed. The wet precipitate is then placed upon a filter, or dried in a dish, and weighed.

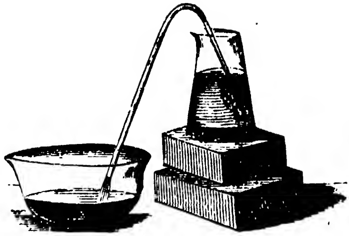


Fig. 78.

Evaporation.

544 The process of evaporation is generally most conveniently effected in Berlin porcelain evaporating basins, either on a sand-bath or over a lamp.* Care must be taken, in quantitative experiments, that no loss is occasioned by spurting, and, on this account, it is safer not to allow the liquid absolutely to boil. When a saline solution has to be evaporated to dryness, it often becomes covered, when concentrated, with a pellicle of solid matter, preventing the escape of the steam, which, being thus confined, occasionally causes some of the mixture to be projected violently from the basin. The best way of avoiding this, is to stir the mixture constantly with a glass rod, from the time when the pellicle begins to form until it is evaporated to dryness. Or the evaporating dish may be loosely covered with a *dial-glass* placed with its convexity downwards, so as to prevent the chilling of the surface and consequent deposition of the solid crust.

545 It is often advisable, and in the case of many liquids, as those containing organic matter, necessary, to evaporate over a water-bath; by this means the heat is never allowed to rise higher than 212° F. For this purpose, a common saucepan, or almost any

* A ring (Argand) gas burner, without a chimney, is exceedingly well adapted for evaporation in quantitative analysis.

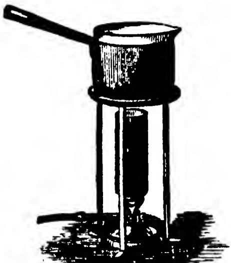


Fig. 79.

vessel used for boiling water in, may be employed, placing the dish containing the solution over the top, as shown in figure 79, so as to expose it to the action of the steam. For the laboratory, a convenient form of water-bath is shown in figure 80; it may be made of copper or zinc plate, and the holes should be fitted with lids, to cover them when not wanted.

546 A convenient method of drying certain substances

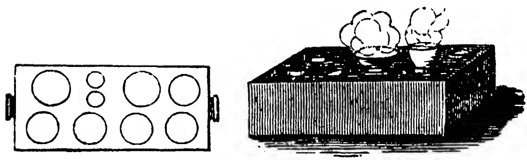
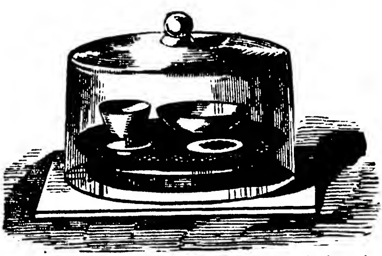


Fig. 80. Hot-Water Bath.

which are liable to decomposition at a slightly elevated temperature, is to place them under the receiver of the air-pump (figure 81), over an open pan of strong sulphuric acid: the latter absorbs the moisture which rises from the substance, and a gradual and complete desiccation may be effected at ordinary temperatures. The dishes containing the substance to be dried may be placed on a sheet of perforated zinc, resting on the pan of acid. If an air-pump is not at hand, the same effect may be produced, though more slowly, by placing the receiver, enclosing the substance and acid, upon a flat piece of glass. Owing to the slowness of the evaporation, this method is



. 81. Desiccation over Sulphuric Acid.

well adapted for obtaining large and well-defined crystals from saline solutions, &c.

547 When a uniform temperature is required, higher than 212° F., it may be obtained by immersing the dish or flask containing the substance to be evaporated in a bath of oil or some saline solution, the boiling point of which is near the desired temperature. Olive oil may be heated to nearly 500° F. (260° C.) without suffering much decomposition, and forms an extremely useful bath for many purposes, since, by regulating the lamp, and placing a thermometer in the oil, any lower temperature can readily be kept up.

A bath of melted paraffin is very convenient for temperatures between 212° F. (100° C.) and 400° F. (204° C.), or even somewhat higher. A temperature of 224° F. (107° C.) may be maintained by means of a saturated solution of common salt, whilst a bath of chloride of calcium or chloride of zinc may be maintained at almost any required temperature, according to the strength of the solution. An air-bath, or metallic box, with a chimney to create a draught, and a few air-holes near the bottom, is most useful for operations at temperatures above the boiling point, especially if provided with an automatic gas-regulator.

Ignition.

548 It is generally necessary, previous to weighing a precipitate, in quantitative analysis, to heat it to redness, in order to insure perfect dryness. This is usually done in a weighed platinum or porcelain crucible, either in a furnace or over a lamp. When the crucible is to be heated in the furnace or open fire, it should be enclosed in one of earthenware, to protect it from contact with the coals and dirt, a little magnesia being interposed between the two (note to 491). If the lid is made of the form shown in figure 82, it may also be used as a capsule, independently of the crucible.

Fig. 82.

549 When gas is available, scarcely any other source of heat is necessary for the purpose. A small platinum crucible may be heated to low redness over the

naked flame, resting on a small wire triangle placed on the top of the chimney (fig. 83).

A mixture of gas and air, however, gives a much more intense heat, owing to the more perfect oxidation of the combustible matter: such a mixture is easily obtained by placing a small piece of wire-gauze over the chimney, and applying a light to the mixture as it rises through the

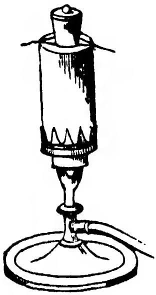


Fig. 83.

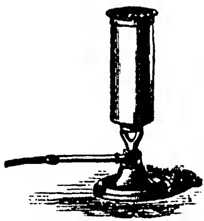


Fig. 84. Gauze Burner.

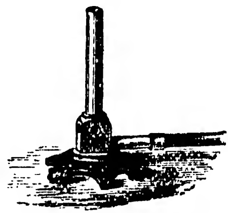


Fig. 85. Bunsen's Burner.

gauze (fig. 84). Bunsen's burner (fig. 85) also furnishes an excellent flame of the mixture of gas with air. The crucible may be supported on a wire triangle.

A gas blowpipe, supplied with air by a double-action bellows, is now to be found in most laboratories. When the crucible is to be raised to a very high temperature it should be surrounded by a jacket of thin iron plate, to prevent loss of heat by radiation.

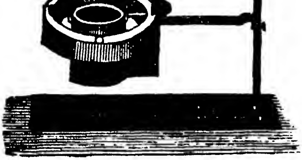


Fig. 86. Rose's Spirit Lamp.

When gas cannot be had, the best lamp for heating a small platinum crucible to redness, is that known as Rose's, the form of which is shown in figure 86; either methylated alcohol or pyroxylic spirit may be burnt in it.*

* The Berzelius spirit-lamp is safer than Rose's, since the reservoir of spirit is not placed so near the flame. A small charcoal fire may also be used with great advantage when gas is not to be had.

Calculation of Results.

550 When the weight of a precipitate has been ascertained, it is necessary to calculate that of the constituent, the weight of which we wish to learn, and this is readily done according to the well-known laws of combination in definite proportions.

For example, let us suppose that we have to determine the percentage of (SO_3) in dry sulphate of soda (Na_2O,SO_3): we dissolve twenty grains of the salt in water, precipitate the sulphuric acid by means of chloride of barium, and weigh the sulphate of baryta thus obtained: from this we have to deduce the weight of the sulphuric acid which it contains; and lastly, to calculate from this the percentage equivalent to it. We find the weight of the sulphate of baryta obtained to be 32·80. Knowing the molecular weight of sulphate of baryta (BaO,SO_3) to be 233, and that of sulphuric acid (SO_3) to be 80, it is easy to calculate how much of the acid is contained in 32·80 grains of the precipitate, thus:—

Mol. wt. of sulph. of baryta.		Mol. wt. of SO_3		Wt. of sulph. of baryta obtained.		Wt. of sulph. acid in 32·80 grs. of sulphate of baryta.
233	:	80	::	32·80	:	$x=11·26$

Thus we find that twenty grains of the dry sulphate of soda contain 11·26 of SO_3; and we have now only to reduce it to a percentage, to complete the calculation, thus:—

20 : 11·26 :: 100 : $x=56·30$ SO_3 in 100 parts of dry sulphate of soda Or, as 20 is the fifth part of 100, the same result may be obtained by simply multiplying by 5. $11·26\times5=56·30$.

The molecular weight of any compound is found by adding together the atomic weights of its component elements, taking as many atoms as are indicated in the formula. Thus, the molecular weight of $BaSO_4$ is $137(Ba)+32(S)+16\times4(O_4)$.

A Table of Atomic Weights will be found in the Appendix.

CHAPTER II.

EXAMPLES IN QUANTITATIVE ANALYSIS.

SECTION I.

Quantitative Analysis of Sulphate of Copper.

551 *Rough weighing of the sample for analysis.*—Cut two equal squares of writing-paper, about two inches across, double them and place them in opposite pans of the rough balance. Place 100 grain weight (or 6·5 grammes*) upon one of them, and into the other, opened out so as to form a gutter, place the crystals of sulphate of copper, until they counterpoise the weight in the opposite scale.

552 *Removal of adhering moisture.*—Reduce the crystals to powder in a mortar, place it between two or three folds of blotting-paper, and press. Repeat this with fresh blotting-paper if necessary, till the salt no longer moistens the paper. Preserve the dried salt in a well-closed dry tube.

553 *Exact weighing of the sample for analysis.*—Weigh a watch-glass (or a light porcelain dish or crucible), and mark the weight upon it (on the glass with a diamond, on the porcelain with ink). Add 20 grains (or 1·5 gramme) to the weights on the scale, and carefully pour the salt from the tube into the watch-glass, until the requisite weight has been taken.†

554 *Estimation of the water of crystallization.*—Weigh a porcelain crucible with its cover, and mark the weight upon each with ink, appending a letter to the weight (thus, A 234·12), in order that the same cover may always be used. Place the crucible upon a

* The grammes corresponding to the weights in grains are here given only in round numbers, to save trouble in counting weights. Tables of Continental Weights and Measures will be found in the Appendix.

† If a very accurate balance be used, it is difficult and tedious to weigh out any given quantity. It is better to place *about* the required quantity in the watch-glass, and to determine the exact weight of the watch-glass and substance, from which the weight of the glass may afterwards be deducted. *Never leave a good balance on the swing whilst the weights are being adjusted. Do not touch the weights with the fingers.* Always arrange the weights in order on the floor of the balance, and count them carefully before restoring them to the box. *Enter the weights in a note-book on the spot.*

half-sheet of smooth paper, and transfer to it the twenty grains of sulphate of copper previously weighed. If any particles adhere to the watch-glass, brush them off with a camel's hair pencil, and return to the crucible any particles which have fallen on to the paper. Place the crucible (uncovered) in the water-oven (fig. 67) for about two hours;* allow it to cool (covered) under the desiccator (fig. 81), and weigh it. Repeat the weighing at intervals of half an hour until no further loss is observed. The loss is then calculated upon 100 parts of the salt.

555 *Estimation of the water of constitution.*—Place the open crucible with its contents, from (554), over the Argand burner (fig. 83), so that it may be heated to about 300° F. (149° C.), for half an hour; cool it under the desiccator, and weigh it; repeat the weighing at intervals of a quarter of an hour till no further loss is observed. Calculate the percentage from the original weight of the sulphate of copper.

556 *Estimation of the sulphuric acid.*—Weigh out 20 grains (or 1·5 gramme) of the sulphate of copper, as in (553), transfer it to a pint beaker, rinsing any adhering portions from the watch-glass with the washing-bottle. Dissolve it, with the aid of heat, in about half a pint of water, add a few drops of hydrochloric acid, and heat the solution nearly to the boiling point (fig. 68), (but without allowing it to bubble). Boil some solution of chloride of barium, and add it gradually to the hot solution of the sulphate,† stirring it with a glass rod, until no further increase in the quantity of the precipitate is observed; allow the precipitate to settle partially, and pour some more chloride of barium carefully into the clear liquid, to make sure that no further precipitate is produced; rinse the glass rod carefully into the beaker, and remove it. Cover the beaker with a dial-glass to exclude dust, and set it aside until the precipitate of sulphate of baryta has settled down, leaving the solution perfectly clear.

* To ascertain whether all the water has been expelled, place a cold glass plate upon the mouth of the crucible; it is useless to weigh as long as any dew is deposited upon it.

† In quantitative analysis, pour the reagent down the side of the vessel, or down a glass rod, to avoid splashing. About 20 grains of crystallized chloride of barium are necessary to precipitate the 20 grains of crystallized sulphate of copper.

557 Decant the clear liquid down a glass rod into another beaker, without disturbing the precipitate (fig. 73), pour about a quarter of a pint of boiling water over the latter, stir it up with a glass rod, and allow it to settle as before. Take a filter-paper about four inches in diameter, make a very neat filter (fig. 20), fit it closely into a good funnel (fig. 72), moisten it with water, and press its sides closely to the funnel. Pass the clear liquid first decanted through this filter (fig. 73), to retain any particles of precipitate which may have been poured away with it; test the filtered liquid with chloride of barium, to be sure that all the sulphuric acid has been removed, and throw it away. Again decant the water from the precipitate in the beaker, replace it by fresh boiling water, and pass the decanted liquid through the filter as before. When the precipitate has again subsided, decant the clear liquid on to the filter, throwing away the filtrate from time to time, so that there may not be much liquid to filter again if any of the precipitate should accidentally pass through the filter. Finally, rinse the precipitate carefully on to the filter with boiling water from a hot water washing-flask (fig. 74), taking care not to fill the filter above one-fourth of its capacity. Use a piece of caoutchouc tube slipped over the end of a glass rod, to remove the last portions of precipitate from the beaker. Wash the precipitate with boiling water (fig. 74), until a drop of the washings, caught upon a slip of glass, and evaporated, leaves no appreciable residue. Dry the precipitate as in (540). Weigh a porcelain or platinum crucible, with its cover, and proceed, as in (541), to calcine the precipitate and incinerate the paper. Weigh the covered crucible with the sulphate of baryta and ash, and calculate the amount of sulphuric acid as in (550).*

558 *Estimation of the oxide of copper.*—Proceed exactly as in (556), but omit the hydrochloric acid, and instead of chloride of barium, add a hot solution of potash, until the precipitate of oxide of copper has a uniform dark brown, nearly black colour.

Conduct the rest of the operation precisely as in (557). A porcelain crucible should be employed for calcining the precipitate, and since this is very liable to absorb

* Never throw precipitates away until the analysis is completed, lest they should be required for ascertaining the cause of any excess or deficiency.

water from the air, the crucible must be allowed to cool under the desiccator, and weighed as quickly as possible. Calculate the proportion of oxide of copper in 100 parts of the original salt.

559 *Statement of the results of the analysis.*—The results should be entered in the note-book in the following manner:—

Water of Crystallization.

Sulphate of copper taken . .	20·00
Crucible and cover	234·12
After 2 hours	248·62
„ $2\frac{1}{2}$ „	248·36
„ 3 „	248·36
	234·12
Dried salt .	14·24
Water lost . .	5·76
Per cent. . . .	28·80

Water of Constitution.

After heating for $\frac{1}{2}$ hour . .	246·96
„ „ $\frac{3}{4}$ „ . .	246·92
„ „ 1 „ . .	246·92
Crucible with salt dried at 212°	248·36
„ „ „ over Argand	246·92
Water lost . .	1·44
Per cent. of original salt . .	7·20

Sulphuric Acid.

Watch-glass and sulphate of copper	89·80
Watch-glass	69·80
Sulphate of copper	20·00
Crucible, cover, and sulphate of baryta	252·72
Crucible and cover	234·12
Sulphate of baryta	18·60
Corresponding to SO_3	6·3862
Per cent.	31·93

Oxide of Copper.

Sulphate taken	20·00
Crucible, cover, and oxide of copper	240·46
Crucible and cover	234·12
Oxide of copper .	6·34
Per cent. . . .	31·70

RESULTS OF ANALYSIS OF SULPHATE OF COPPER.

	Found.	*Calculated* ($CuO.SO_3,H_2O, 4Aq.$)
Oxide of copper	31·70	31·85
Sulphuric acid (SO_3) . . .	31·93	32·07
Water of constitution . .	7·20	7·22
Water of crystallization .	28·80	28·86
	99·63	100·00

Atomic weights employed, Cu=63·4, O=16, S=32, H=1.

SECTION II.

Quantitative Analysis of Chloride of Sodium.

560 Weigh roughly (551) 50 grains (or 3 grammes) of the salt, and heat it for ten minutes in an open porcelain or platinum crucible over an Argand burner (fig. 83). Allow it to cool partly in the covered crucible and transfer it, when still warm, to a well closed dry tube.

561 *Estimation of the sodium.*—Weigh accurately (553) 20 grains of the salt (1·5 gramme); place it in a large porcelain crucible which has been previously weighed, with its cover (554), pour upon it about half a measured ounce of water, add gradually about twenty drops of strong sulphuric acid, stirring with a thin glass rod; rinse the rod into the liquid, remove it, and place the open crucible on a steam-bath (fig. 79). When the evaporation has proceeded as far as possible, place the crucible on a triangle over an Argand burner with a very small flame, and continue the evaporation. If any solid crust forms over the surface of the liquid, cover the crucible, leaving a crevice for the escape of vapour. When white fumes of sulphuric acid are observed, the crucible is uncovered, and the heat gradually increased till they are no longer visible, and the sulphate of soda in the crucible is quite dry. The cover is dried by gradually heating it, and placed on the crucible, which is allowed to cool, and weighed. A small fragment of carbonate of ammonia is then introduced (to decompose any traces of bisulphate of soda which might be left), the crucible covered, and the calcination and weighing repeated, the crucible being uncovered for a minute or two at the close of the calcination. Repeat these operations till a constant weight is obtained.

Calculate the weight of the sodium from that of the sulphate of soda; suppose that 24 grains of the latter have been obtained.

$$\underbrace{142}_{Na_2SO_4} \; : \; \underbrace{46}_{Na_2} \; :: \; \underbrace{24}_{\text{Wt. of sulph. soda obtained.}} \; : \; \text{Sodium in 20 grs. of salt employed.}$$

562 *Determination of the weight of a dried filter.*—Select a beaker (about $2\frac{1}{2}$ inches by $1\frac{3}{4}$ inches) and cover it with a watch-glass, which should fit pretty closely, with the convexity downwards. Weigh them together, and mark the weight upon both with a diamond (554). Their joint weight should not exceed 450 grains (30 grammes). Fold a circular filter, about three inches in diameter, and place it in the water-oven* (fig. 67) for an hour: introduce it while warm into the weighed beaker, cover it with the watch-glass, and weigh. Replace the filter in the water-oven, for a quarter of an hour, and weigh again. Repeat this till a constant weight is obtained, and note its weight upon the edge of the filter with a light pencil mark.

563 *Estimation of the chlorine.*—Weigh accurately (553) 10 grains (or 0·5 gramme) of the dried salt, transfer to a half-pint beaker, and dissolve in about three measured ounces of water. Heat the solution, add a few drops of nitric acid, and add solution of nitrate of silver, constantly stirring, as long as the quantity of the precipitate seems to increase. Stir briskly with a glass rod, to encourage the separation of the precipitate, and add a little more nitrate of silver to the clear liquid, to be sure that all the chlorine is precipitated.† Leave the glass rod in the beaker, and set it aside till the fluid is clear or nearly so; the separation of the precipitate being promoted, if necessary, by heat and stirring. Filter the liquid through the weighed filter (562) upon which every particle of the precipitate must be carefully rinsed with hot water, wash with hot water till a few drops of the washings give no turbidity with hydrochloric acid. Dry the filter as in (540) till it can be safely lifted into the weighed beaker (562), place the latter open in the water-oven for an hour, cover it with the watch-glass, allow it to cool under the desiccator (fig. 81), and weigh. Repeat the weighing at intervals of twenty minutes, till a constant weight is obtained. Deduct the weight of the beaker, watch-glass, and filter, and calculate the weight of the chlorine from that of the

* When a water-oven is not at hand, filters may be dried in a beaker suspended in a vessel of boiling water by means of a perforated metal plate through which the beaker passes exactly.

† About 30 grains of (solid) nitrate of silver are required to precipitate the 10 grains of salt.

chloride of silver.* Suppose 24·5 grains of chloride of silver to be obtained.

		Cl		Wt. of chlor. silver obtained.		Wt. of chlorine found.
143·5	:	35·5	::	24·5	:	x

RESULTS OF THE ANALYSIS OF COMMON SALT.

	Found.	*Calculated* (NaCl).
Sodium	. . . . . .	39·32
Chlorine	. . . . . .	60·68
		100·00

SECTION III.

Quantitative Analysis of Carbonate of Lime.†

564 Weigh roughly (551) 50 grains (or 3 grammes) of the carbonate of lime, and heat it, very moderately, in a crucible (555), until dew is no longer deposited on a glass plate held over it. Cool it under the desiccator (fig. 81) and transfer it to a well closed dry tube.

565 *Estimation of the lime.*—Weigh accurately (553) 20 grains (or 1 gramme), place it in a pint beaker, and add about ¼ pint of water. Cover the beaker with a dial-glass, convexity downwards, slip it aside, leaving a crevice, and pour nitric acid, *very gradually*, down the side of the beaker, waiting till the effervescence has subsided before adding a fresh portion. When the carbonate is completely dissolved, place the covered beaker over a lamp (fig. 68), and gradually heat as long as any bubbles of carbonic acid are observed. Rinse the dial-glass into the

* The weight of the chloride of silver may be obtained more rapidly (though not so certainly by beginners) without using a weighed filter. It is then unnecessary to dry in the water-oven, but when the precipitate has been dried as in (540), it is transferred from the paper into a weighed porcelain crucible, heated till it fuses, and weighed. The filter, with the minute portion of adhering precipitate, is incinerated as in (542), and the ash weighed. The small portion of chloride adhering to the paper becomes reduced to metallic silver by the hydrogen of the latter. This amount of silver is multiplied by 1·33 to convert it into chloride of silver, which is added to the weight of the larger portion; or if it be preferred, it may be weighed as chloride, by heating, first with a drop of nitric, then with a drop of hydrochloric acid, and drying in the crucible.

† Either Iceland spar, or pure precipitated carbonate of lime (prepared chalk) is best for this purpose. White marble or chalk, not being quite pure, will give less accurate results. Iceland spar or marble will not require drying, but the others should be dried in the water-oven or over a very low flame.

beaker and add ammonia, very gradually, stirring with a glass rod, until a drop of the liquid decidedly blues reddened litmus.* Add oxalate of ammonia as long as it appears to increase the precipitate.† Heat the mixture to boiling, allow the precipitate to settle; test the supernatant liquid with a little more oxalate of ammonia; pour it through a filter (about 4½ inches in diameter), collect the precipitate upon the filter; adopting the precautions mentioned in (557), wash it with hot water till the washings no longer leave any important residue when evaporated on a slip of glass. Dry the oxalate of lime upon the filter (540), and proceed further as in (542). During the calcination of the oxalate of lime, it becomes converted into carbonate (with evolution of carbonic oxide), but a part of the carbonate is usually converted into caustic lime; hence, previously to weighing, sprinkle the calcined precipitate, after cooling, with a little powdered carbonate of ammonia, moisten it with water, and dry it at a very moderate heat. After weighing it, repeat the treatment with carbonate of ammonia, until the weight is constant.‡ Calculate the amount of lime from that of the carbonate obtained. Suppose the latter to weigh 19·85 grains.

Mol. wt. of carb. lime.		Mol. wt. of lime.		Wt. of carb. lime obtained.	Wt. of lime found.
100	:	56	::	19·85	

566 *Estimation of the carbonic acid.*—Fit up the apparatus represented in (fig. 87), taking great care that the joints are perfectly air-tight, and that the

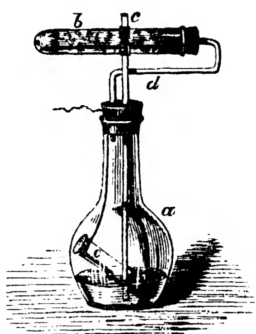


Fig. 87.

* A little alumina or silica or (brown) peroxide of iron may possibly be precipitated here, and if the quantity be of any importance, should be filtered off.

† About 30 grains of crystallized oxalate of ammonia are required to precipitate the lime from 20 grains of carbonate.

‡ If it be preferred, the carbonate of lime may be entirely converted into caustic lime by very strong ignition over a gas-blowpipe (best in a platinum crucible) until its weight is constant.

whole apparatus is as light as possible. The flask (*a*) should be capable of containing about four ounces of water. The tubes *c* and *d* may be very narrow; $\frac{1}{8}$ inch will suffice for their outside diameter; their edges should be rounded with the blowpipe flame before passing them through the cork; *c* passes down to the bottom of the flask. The drying tube *b* may be made out of a light broken test-tube, about $\frac{1}{2}$ inch in diameter, drawn out to a narrow neck, and cut off so as to leave the point open; the edges should be rounded off in the flame. This tube should be about $3\frac{1}{2}$ inches long.*

Put a loose plug of cotton wool into the wide end of the tube, and thrust it into the narrow end; fill the tube with fragments of (porous, not fused) chloride of calcium as large as small peas, insert another loose plug of cotton wool before attaching the tube to the perforated cork on *d*. The tube *e* should be about $1\frac{1}{2}$ or 2 inches long, and may be made from a broken test-tube.

Weigh this tube, add 20 grains (or 1 gramme) to the weights, introduce more than that quantity of the dried carbonate of lime (564) into the tube, and empty out small quantities until the weight is correct.

Pour into the flask six drachms of strong hydrochloric acid and six drachms of water; attach the tube *e* to a horsehair, and sling it in the flask, securing the hair between the cork and the glass, so that the tube containing the carbonate may hang safely above the surface of the acid while the whole apparatus is being accurately weighed. After the exact weight has been recorded, close the tube *c* with a piece of vulcanized tubing stopped with glass rod; gradually incline the flask so as to allow the acid to act upon the carbonate; the carbonic acid is disengaged with effervescence; is deprived of moisture by the chloride of calcium, and leaves the apparatus lighter than before. When the effervescence has ceased, and all the carbonate is dissolved, *gently warm* the flask, to expel the carbonic acid from the liquid, remove the stopper from the tube *c*, fit a cork or a piece of vulcanized tube on to the end of *b*, to avoid wetting it with the lips, and draw air slowly through the apparatus, to displace all the car-

* A piece of light tubing with a cork and small piece of tube, instead of the narrow neck, may be used, if the sharp edges be rounded so as not to cut the corks. Or a hole may be blown at the end of a short test-tube, as in fig. 87.

bonic acid. Weigh the apparatus and suck air through it again, till no further loss of weight is observed. The total loss of weight is that of the carbonic acid expelled.

RESULTS OF THE ANALYSIS OF CARBONATE OF LIME.

	Found.	*Calculated* ($CaO.CO_2$).
Lime (CaO) . . .	. . . . .	56·00
Carbonic acid (CO_2) .	. . . . .	44·00
		100·00

SECTION IV.

Quantitative Analysis of Sulphate of Magnesia.

567 Weigh roughly (551) 100 grains (or 6·5 grammes) of the crystallized salt, remove the adhering moisture as in (552), and preserve the dry salt in a corked dry tube.

Estimate the *water of crystallization* in 20 grains of the salt, as in (554), using an air-bath heated to 300° F. (150° C.) instead of a water-oven, and afterwards estimate the *water of constitution* in the same portion.

568 *Estimation of the magnesia.*—Weigh accurately (553) 20 grains (or 1 gramme) of the salt, place it in a half-pint beaker, and dissolve it in about two ounces of water. Add enough solution of chloride of ammonium to prevent the precipitation of any magnesia on adding ammonia,* which is then to be added in considerable excess. Phosphate of soda is next added, as long as it appears to produce a precipitate,† and the solution is briskly stirred with a glass rod, without rubbing the sides of the beaker; the glass rod may be left in the beaker. When the precipitate has subsided, add a few drops more phosphate of soda to the clear liquid, to be quite sure that no further precipitate is produced: cover the beaker, and set it aside for at least twelve hours. Collect the precipitate upon a filter about 4 inches in diameter, using water containing one-eighth of its bulk of strong ammonia for rinsing the precipitate on to the filter (557) as well

* About 10 grains of solid chloride of ammonium will be sufficient for this purpose.

† About 30 grains of crystallized phosphate of soda dissolved in water will be required.

as for subsequently washing it until the washings, after being acidulated with nitric acid, give but a faint turbidity with nitrate of silver. Dry (540) the filter with the precipitate; transfer (542) as much of the latter as possible, to a porcelain or platinum crucible which has been weighed with its cover, and heat it gradually in the loosely covered crucible (fig. 83), finally raising the temperature to a red heat. In this way, the precipitate of phosphate of magnesia and ammonia ($MgNH_4PO_4$) is converted into pyrophosphate of magnesia ($Mg_2P_2O_7$), which is allowed to cool and weighed. It may then be emptied out of the crucible (preserving it for subsequent examination if the result should prove incorrect), and the filter may be incinerated as in (542), the resulting weight, *minus* the ash of the filter itself, being added to that of the larger portion of the ignited precipitate.* The composition of the latter being $2MgO,P_2O_5$, or $Mg_2P_2O_7$, the amount of magnesia is calculated by the following proportion; in which the weight of the calcined precipitate is supposed to be 9 grains:—

Mol. wt. of pyrophosph. of magnesia.		Two mol. wts. of magnesia.		Wt. of ignited precipitate.		Wt. of magnesia found.
222·6	:	80·6	::	9·00	:	x

569 Estimate the *sulphuric acid* in 20 grains (or 1 gramme) of the salt according to (556).

RESULTS OF THE ANALYSIS OF SULPHATE OF MAGNESIA.

	Found.	*Calculated* ($MgO.SO_3,H_2O$, 6Aq.)
Magnesia (MgO)		16·26
Sulphuric acid (SO_3)		32·52
Water of constitution		7·32
Water of crystallization		43·90
		100·00

SECTION V.

Quantitative Analysis of Arsenious Acid.

570 *Estimation of the arsenic.*—Weigh accurately (553) 5 grains (0·3 gramme) of powdered arsenious acid; transfer to a pint beaker, add an ounce or two of

* The above mode of proceeding is recommended, because the filter is very difficult to burn in this case, from its retaining traces of phosphate, and it can scarcely be heated sufficiently to burn off the carbon on the top of the mass of precipitate.

water, warm it, and add potash in small quantities until the arsenious acid is entirely dissolved. Add about four ounces more water and enough hydrochloric acid to render the liquid slightly acid. Cover the beaker with a dial-glass having a notch cut in it for the passage of the delivery-tube from the sulphuretted hydrogen apparatus. Pass the sulphuretted hydrogen gas through the solution in the covered beaker, until, after disconnecting the tube from the gas-bottle, uncovering the beaker, and blowing off the gas from the space above the liquid, a distinct smell of sulphuretted hydrogen is perceived on stirring the latter with the tube. Rinse any splashings off the dial-glass into the beaker. Weigh a filter (about 4 inches in diameter) which has been dried in the water-oven (562), and collect the bright yellow precipitate of sulphide of arsenic upon it, using the tube instead of a stirring rod to guide the liquid on to the filter.* Wash the precipitate till the washings are no longer precipitated by nitrate of silver, and proceed with the drying as in (563).† Calculate the weight of arsenic present from that of the sulphide of arsenic (As_2S_3) obtained. Suppose the latter to weigh 6·2 grains.

$$\underbrace{246}_{As_2S_3} : \underbrace{150}_{As_2} :: \underbrace{6{\cdot}2}_{\text{Wt. of sulph. arsenic obtained.}} : \underbrace{x}_{\text{Wt. of arsenic found.}}$$

571 *Estimate the oxygen by difference—i.e.* by deducting the weight of the arsenic from that of the arsenious acid.

RESULTS OF THE ANALYSIS OF ARSENIOUS ACID.

	Found.	*Calculated* (As_2O_3).
Arsenic		. . . . 75·75
Oxygen		. . . . 24·25
		100·00

* Should there be any difficulty in removing the last portions of the precipitate from the beaker or tube with a caoutchouc cap (or a tuft at the end of a feather), rinse them with a very little water containing a drop of ammonia, which will dissolve the sulphide of arsenic, and reprecipitate this by acidifying the liquid with hydrochloric acid.

† Two or three hours will probably be required for drying the sulphide of arsenic.

SECTION VI.

Volumetric Analysis. Acidimetry.*

572 Weigh roughly (551) 100 grains (or 6 grammes) of pure† bicarbonate of soda ($NaHCO_3$), place it in a platinum or porcelain crucible, and heat it for a few minutes over a gauze or Bunsen burner. (If a platinum crucible be used, the Argand burner will be sufficient.) Cool the covered crucible under the desiccator (546) and weigh. Repeat the heating till the weight is constant, and preserve the carbonate of soda (Na_2CO_3) in a well-closed and thoroughly dry tube.

573 Measure out 100 grains (or 5·4 cubic centimetres) of strong sulphuric acid (H_2SO_4) in a pipette; introduce it into a measuring flask containing a little water, and make it up to 7000 grains (or 500 cubic centimetres = $\frac{1}{2}$ litre) with water. Preserve the diluted acid in a stoppered bottle.

These measures are only given as a guide, so that the acid may not be made of any unreasonable strength, and may be adapted for other uses. If more accurate measures are not at hand, ordinary druggists' measures may be used; 100 minims of sulphuric acid being mixed with 16 ounces of water. Pipettes (Fig. 88) are sold, which deliver 100 grain measures when filled to a certain mark on the stem. A 10 c. c. pipette would also answer the purpose. It is not advisable to suck the acid into the pipette with the mouth. The acid should be poured into a cylindrical vessel in which it will stand high enough to fill the pipette up to the mark on immersion. Measuring-flasks are sold, which contain 7000 grains ($\frac{1}{10}$ gallon = a *decigallon*) when filled up to a certain mark on the neck. They are also made for the fractions of a litre. Accurate measures may be made by the student by introducing known weights of water into the vessels, and marking the level with a diamond or hard steel point. The Continental scale is the most convenient, because 1 *c. c. of distilled water at* 4° C. (39°·2 F.) *weighs one gramme.* The narrower the vessel at the point where the mark is made, the more exact will be the measurement.

Fig. 88. Pipette (100 grs).

574 *Estimation of the amount of sulphuric acid in the diluted acid.*—Weigh accurately (553) 10·6 grains‡ (or ·53 grm.) of the pure car-

* By volumetric analysis is understood the method of determining the quantity of a substance by the use of a measured volume of a reagent of known strength.

† The bicarbonate purchased at the druggist's is often almost chemically pure. It should be tested for sulphate and chloride of sodium.

‡ This quantity is taken as being a convenient fraction of the molecular weight of the carbonate, which is 1·06.

bonate of soda (572); transfer it to a pint flask, rinsing in the adhering particles with water, and dissolve it in about four ounces of water. Add enough solution of litmus to colour the liquid distinctly blue. (It is well to measure the litmus roughly, using the same quantity in all cases.)

Fill a *burette* (576) with the diluted acid, and after measuring it exactly, add it, in small portions at a time, to the solution of carbonate of soda in the flask, with constant shaking, until the blue colour has changed to purple (due to the action of carbonic acid upon the litmus). This will happen when somewhat more than half the requisite quantity of acid has been added. Place the flask upon a piece of wire gauze over a lamp (fig. 89), and boil the solution to expel the carbonic acid and restore the blue colour. Continue the cautious addition of the acid, with occasional boiling, until the colour of the litmus has been changed to a light red which is permanent even on boiling for some minutes, showing that the acid has been added in slight excess. Note carefully the number of measures of acid, and calculate the quantity of sulphuric acid present. Suppose that 680 grains of acid have been used.

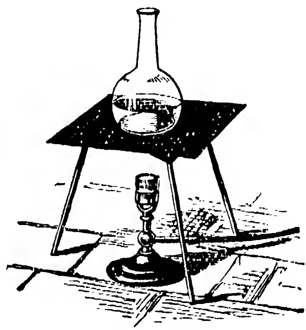


Fig. 89.

The 10·6 grains ($\frac{1}{10}$th molecule) of carbonate of soda employed, would require 9·8 grains of hydrated sulphuric acid ($H_2O.SO_3$) or $\frac{1}{10}$th molecule, to neutralize them. Hence this quantity was present in the 680 grain measures of acid employed.

Calculate the quantity of sulphuric acid in 1000 grain measures (or in 100 cubic centimetres), and record it upon the label of the bottle.

The foregoing experiment should be repeated, when the result should be almost identical. It may be confirmed by estimating the acid in a measured quantity of the diluted acid, as in (556).

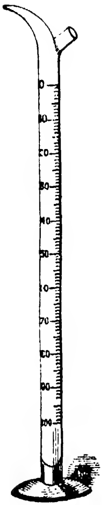


Fig. 90.

Fig. 91.

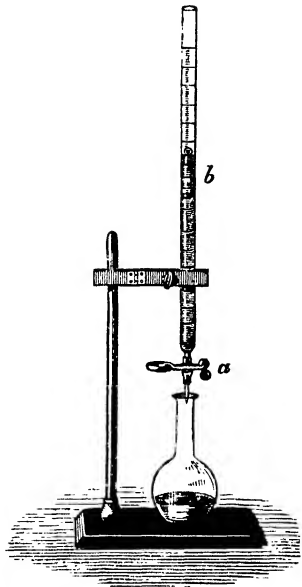


Fig. 92.

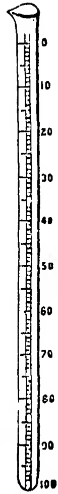


Fig. 93. Alkalimeter Tube.

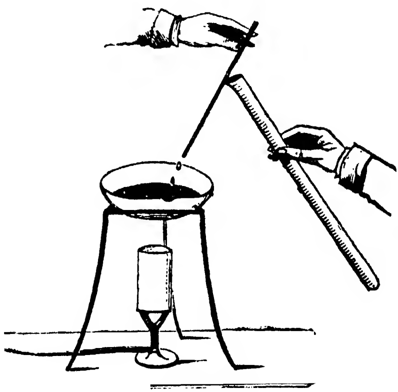


Fig. 94.

575 The quantity of acid in samples of diluted hydrochloric and nitric acids may be estimated in a similar manner.

576 A *burette* is a carefully divided tube from which a liquid may be poured in very small portions at a time. One of the simplest and most convenient is that represented in fig. 90; the flow of the liquid through the narrow orifice is checked by the pressure of the finger upon the wider opening. Such a burette

is most conveniently filled with a large pipette (fig. 91). This is plunged into the liquid, contained in a Winchester quart bottle, which is allowed to fill it; the upper end is then firmly closed with the finger, the tube withdrawn from the bottle, its lower opening held over the wider opening of the burette, and the finger removed, when the liquid flows out into the burette.

Mohr's burette (fig. 92), from which the liquid is allowed to flow by opening the *nipper-tap* a, allows of more exact measurement, especially if a float b, made of a glass tube slightly weighted be placed in it. A circular mark is made round the float with a file or a diamond, and the measurements are made by observing which of the divisions on the burette exactly coincides with this line. A burette of this kind with a glass stopcock, though somewhat more expensive, is far more useful.

An ordinary alkalimeter tube (fig. 93) gives very good results if carefully employed, the acid being then poured down a glass rod into the alkaline solution in a dish or beaker (fig. 94).

SECTION VII.

Alkalimetry.

577 *Estimation of the useful soda in a sample of crystallized carbonate of soda* ($Na_2CO_3.10H_2O$).—Prepare some *standard sulphuric acid* according to (573) and determine its exact strength by (574). Weigh accurately (553) 25 grains (or 1·5 gramme) of the carbonate of soda, and proceed exactly as directed in (574).

Calculate the amount of soda by the following proportions:—

$$1000 : \left\{\begin{array}{c}\text{Wt. of sulph. acid} \\ (H_2SO_4)\text{ contained} \\ \text{in 1000 grs. of the} \\ \text{standard acid.}\end{array}\right\} :: \left\{\begin{array}{c}\text{No. of grain mea-} \\ \text{sures of standard} \\ \text{acid used in this} \\ \text{experiment.}\end{array}\right\} : x \left\{\begin{array}{c}\text{Wt. of } H_2SO_4 \\ \text{required to} \\ \text{neutralize the} \\ \text{soda.}\end{array}\right.$$

$$\underbrace{\begin{array}{c}\text{Mol. wt. of} \\ H_2SO_4\end{array}}_{98} : \underbrace{\begin{array}{c}\text{Mol. wt. of soda} \\ (Na_2O)\end{array}}_{62} :: \left\{\begin{array}{c}\text{Wt. of } H_2SO_4 \\ \text{required to neu-} \\ \text{tralize the soda.}\end{array}\right\} : y \left\{\begin{array}{c}\text{Wt. of soda } (Na_2O) \\ \text{present in the} \\ \text{sample.}\end{array}\right.$$

Calculate the weight of soda in 100 grains of the sample. Perfectly pure crystallized carbonate of soda yields 21·68 per cent. of soda.

578 Estimate the potash in a sample of crystals of bicarbonate of potash, as in (577), substituting the molecular weight of potash (K_2O=94·2) for that of soda in the calculation. Perfectly pure crystallized bicarbonate of potash ($KHCO_3$) yields 47 per cent. of potash.

579 Estimate the potash in 100 grain measures (or 10 cubic centimetres) of the solution of potash in use in the laboratory, by diluting it with water, adding litmus, and conducting the process as in (574).

580 Estimate the ammonia in the solution of ammonia in use in the laboratory. Introduce 1000 grain measures of the standard sulphuric acid (573) into a beaker, and colour it distinctly red with litmus. Measure 100 grains (or 10 cubic centimetres) of the solution of ammonia in the burette, dilute to 1000 grains (or 100 cubic centimetres) with water, mix by agitation, and add the solution, very gradually, to the acid, with constant stirring, until the litmus just assumes a blue shade. Observe the number of measures used and calculate the quantity of ammonia present thus :—

$$\underbrace{H_2SO_4}_{98} : \underbrace{2NH_3}_{34} :: \left\{\begin{array}{c}\text{Wt. of } H_2SO_4 \\ \text{in 1000 grains} \\ \text{of standard} \\ \text{acid.}\end{array}\right\} : \underbrace{\begin{array}{c}\text{Wt. of ammonia in the} \\ \text{amount of solution used.}\end{array}}_{x}$$

$$\left\{\begin{array}{c}\text{No. of gr. mea-} \\ \text{sures of solu-} \\ \text{tion of am-} \\ \text{monia used.}\end{array}\right\} : \left\{\begin{array}{c}\text{Wt. of am-} \\ \text{monia con-} \\ \text{tained in} \\ \text{them } (x)\end{array}\right\} :: 1000 : \underbrace{\begin{array}{c}\text{Wt. of ammonia in 100 grain mea-} \\ \text{sures of the original solution.}\end{array}}_{y}$$

SECTION VIII.

Estimation of Iron in Red Hœmatite Ore (Fe_2O_3).

581 Weigh roughly (551) 100 grains (or 6 grammes) of the ore, reduce it to an impalpable powder (526), and preserve it in a corked tube. Weigh accurately 5 grains (or ·3 gramme), introduce it into a four-ounce flask, and heat it (fig. 89) with about $\frac{1}{2}$ oz. measure (or 200 grain measures, or 10 cubic centimetres) of strong hydrochloric acid, and one-third as much water, until it is either entirely dissolved, or only a little white silica is left.*

582 *Preparation of a standard solution of potassium permanganate* ($KMnO_4$).—Weigh out (553) 16 grains† (or 1 gramme) of the crystallized permanganate, introduce it into a half-pint flask or beaker, and dissolve it in about 4 measured ounces of water, employing a gentle heat (fig. 89) if necessary. Pour the solution carefully into a measure-flask (573) capable of containing 7000

* This often requires two or three hours, and it may be found necessary to add a little more HCl to replace that which evaporates.

† $\frac{1}{10}$th molecular weight of $KMnO_4$ would be 15·81 grains, and if the purity of the salt can be relied on, calculation would be facilitated by employing this quantity.

grains (or 10,000 grains, or ½ litre); rinse the adhering solution out of the vessel with a little water, and fill the flask up to the mark with water. (If a measure-flask be not at hand, use a druggist's measure, and make the solution up to 16 oz.) Preserve the solution in a stoppered bottle.

583 *Determination of the strength of the solution of permanganate.*—Weigh accurately 3·5 grains (or 0·2 gramme) of clean iron wire (*annealed iron wire*), introduce it into a half-pint flask furnished with a cork and a short piece of narrow glass tube; pour upon it about an ounce (or 500 grain measures, or 30 cubic centimetres) of diluted sulphuric acid (containing one-fifth of its bulk of the strong acid). Apply a gentle heat (fig. 89) till the iron is dissolved. Nearly fill the flask with water (which has been boiled to expel air and allowed to cool), pour the solution very carefully into a pint beaker, rinse the flask twice with water, dilute the solution with water to about half a pint, and add the solution of permanganate, gradually, from a burette,* with constant stirring, until a faint pink tinge is perceptible throughout the whole liquid, proving that all the ferrous sulphate is converted into ferric sulphate, and that the permanganate has been added in slight excess. (About 875 measures of a solution of the above strength would be required.) Observe the number of measures of the solution of permanganate employed, and calculate the quantity of iron to which 1000 grain measures (or 100 cubic centimetres) of the solution correspond, and record it upon the label of the bottle.

The whole operation should be repeated, with a fresh portion of iron, to prove the correctness of the result.

584 It will often be found convenient, in volumetric analysis, to dissolve a weighed quantity of the substance, and to make up the volume of the solution to a definite amount, of which fractions may be taken for several determinations. Thus, in (583), 10·5 grains of iron may be dissolved, the solution made up with water to 1000 grains, and ⅓ of this taken for each experiment (or 1 gramme may be dissolved, made up to 500 c. c. and 100 c. c. taken in each case). In (581) 20 grains of hæmatite may be dissolved, the solution made up to 1000 grains, and 250 grains taken for each experiment (or 1·5 gramme may be dissolved, made up to 500 c. c. and 100 c. c. taken in each case).

* If Mohr's burette be employed, it must have a glass stopcock, since the permanganate acts upon the caoutchouc.

585 The action of the potassium permanganate in the foregoing process is thus explained. When the iron is dissolved in sulphuric acid, ferrous sulphate and hydrogen are produced. $Fe+H_2SO=H_2+FeSO_4$. When the ferrous sulphate and the excess of sulphuric acid are brought in contact with the permanganate,

$$10FeSO_4+8H_2SO_4+2KMnO_4=5Fe_2(SO_4)_3+2MnSO_4+K_2SO_4+8H_2O.$$

586 *Reduction of the ferric chloride* (perchloride of iron) *to the state of ferrous chloride* (protochloride of iron).—To the flask containing the solution of the hæmatite ore in hydrochloric acid (581), attach a perforated cork carrying a short piece of narrow glass tubing; dilute the solution with a little water and add a few fragments of pure zinc.* Heat moderately, adding more zinc if necessary, until the yellow colour of the solution has changed to a pale green. Nearly fill the flask with water (free from air), pour the solution into a pint beaker, rinse the flask twice with water, dilute the solution to about half a pint, add about an ounce (20 cubic centimetres) of dilute sulphuric acid, and proceed with the addition of the standard solution of permanganate of potash as in (583).

Calculate the weight of iron present, by the following proportion:—

$$\underbrace{\text{Gr. measures of permanganate}}_{1000} : \left\{\begin{array}{c}\text{Wt. of iron corre-}\\ \text{sponding to 1000}\\ \text{grs. of the standard}\\ \text{permanganate.}\end{array}\right\} :: \left\{\begin{array}{c}\text{No. of gr. mea-}\\ \text{sures of per-}\\ \text{manganate}\\ \text{employed.}\end{array}\right\} : x \left\{\begin{array}{c}\text{Wt. of iron}\\ \text{in the ore}\\ \text{employed.}\end{array}\right.$$

Pure peroxide of iron would contain 70 per cent. of iron.

587 The reduction of ferric chloride to ferrous chloride by the action of zinc is explained in this equation; $Fe_2Cl_6+Zn=2FeCl_2+ZnCl_2$. The sulphuric acid is subsequently added to convert the ferrous chloride into ferrous sulphate, because the ferric chloride produced by the action of the permanganate upon the former, has a strong yellow colour which would disguise the pink colour indicating the termination of the experiment.

588 The following method of reducing the ferric chloride is an excellent one if carefully executed. Place the flask containing the solution of ferric chloride over a lamp, and add solution of sodium carbonate by degrees till the free acid is neutralized, which may be known by the difficulty in re-dissolving the precipitate produced by the carbonate. Then add solution of sodium sulphite drop by drop till the liquid is nearly colourless. Should any

* The zinc must be tested by dissolving a piece of it in hydrochloric acid, diluting the solution largely with water, and adding *a drop* of permanganate which should not be bleached. If the zinc is not pure, the quantity employed for reduction must be weighed, an equal quantity dissolved in hydrochloric acid, the solution largely diluted, and the amount of permanganate which it bleaches carefully determined. Or the method of reduction with sodium sulphite may be employed instead of that with zinc (588).

precipitate be produced, add a drop or two of hydrochloric acid till it becomes clear. When the liquid is nearly colourless, add some dilute sulphuric acid, and boil until there is no more smell of sulphurous acid; this should not require more than ten minutes. Dilute with water, and proceed to add the permanganate as in 583. The reduction is explained by the equation.

$$Fe_2Cl_6 + H_2O + Na_2SO_3 = 2FeCl_2 + 2HCl + Na_2SO_4.$$

589 *Preparation of a standard solution of bichromate of potash.*—Weigh roughly (551) 50 grains (or 3 grammes) of pure bichromate of potash. Reduce it to powder, and heat it in a porcelain crucible over an Argand burner (fig. 83) for a few minutes. Allow it to cool under the desiccator (fig. 81), and weigh out accurately (553) 29·5 grains (or 2·95 grammes) of it. Dissolve it exactly as directed in (582).

590 *Verification of the standard solution of bichromate of potash.*—Weigh accurately 4 grains (or 0·5 gramme) of iron wire, and dissolve it as directed in (583). Dilute the solution with boiling water, transfer it to a pint beaker, rinse the flask twice, dilute the solution with boiling water to about half a pint, and add the standard solution of bichromate gradually, from a burette, with constant stirring, until a drop taken upon a glass rod, and dropped into a spot of a dilute solution of ferricyanide of potassium,* upon a white plate, gives a clear brown tinge, without any blue or green, showing that all the ferrous salt has been converted into ferric salt by the oxygen of the chromic acid.

Calculate the quantity of iron to which 1000 grains (or 100 cubic centimetres) of the solution of bichromate correspond, and record the result upon the label of the bottle.

591 *Determination of iron in hæmatite by the bichromate process.*—Dissolve 6 grains (or 0·8 grm.) of the finely powdered ore in hydrochloric acid as in (581). Reduce the ferric chloride to ferrous chloride as in (586 or 588), but dilute the solution to half a pint only, and proceed to determine the quantity of bichromate solution required to oxidize it as in (583). Calculate the weight of iron present from the number of measures employed.

* This must be quite free from ferrocyanide. Take a crystal of the red prussiate of potash, wipe it well with a damp cloth, weigh it roughly, and dissolve it in about forty times its weight of water. The solution will contain some ferrocyanide if kept, especially when exposed to the light.

592 The action of the potassium dichromate upon ferrous sulphate in the presence of sulphuric acid results in the production of ferric and chromic sulphates.

$$K_2O.2CrO_3+6FeSO_4+7H_2SO_4=K_2SO_4+3Fe_2(SO_4)_3+Cr_2(SO_4)_3+7H_2O.$$

Hence the molecular weight (295·2) of the dichromate corresponds to 6 atoms (336) of metallic iron.

SECTION IX.

Valuation of Manganese Ore (Peroxide of Manganese).

593 To a pint flat-bottomed flask, adapt, air-tight, a perforated cork fitted with a narrow glass tube about 20 inches long, bent twice at right angles, the shorter limb which passes through the cork being about 3 inches long, and the other about 12 inches. Weigh out, separately and exactly (553) 10 grains (or ·8 gramme) of iron wire (*annealed*), and 10 grains (or ·8 gramme) of the *very finely powdered* manganese ore. Place the iron wire in the flask, pour upon it three measured ounces (or 1500 grains, or 90 cubic centimetres) of diluted sulphuric acid (containing one measure of oil of vitriol and three measures of water); attach the cork with the bent tube, and allow the long limb to dip down to the bottom of a 4-oz. flask half full of distilled water. Apply a gentle heat until all the iron has dissolved; remove the cork, and introduce the 10 grains of manganese ore; boil gently (with the tube still dipping into water) until all the black oxide of manganese is dissolved;* remove the lamp, so that the steam may condense, when the pressure of the air will force the water from the smaller into the larger flask, and thus anything which has splashed over will pass back into the flask; pour a little more water into the small flask to rinse it, and allow it to go back in the same way. Remove the cork, add enough distilled water to make the solution up to about half a pint, and ascertain how much iron has been left unconverted into ferric oxide by the available oxygen of the peroxide of manganese; this is effected by a standard solution either of permanganate of potash, as in (583) or of bichromate, as in (590).

Deduct the weight of iron which is found unconverted, from the 10 grains employed, and calculate the weight of

* This often requires two or three hours. If the ore were very rich, it might require the addition of a little more iron, which may be weighed and added. 10 grains of pure MnO_2 would require 12·8 grains of iron.

peroxide of manganese present by the following proportion :—

$$\underset{112}{Fe_2} : \underset{87}{MnO_2} :: \left\{\begin{matrix}\text{Wt. of iron}\\ \text{oxidized.}\end{matrix}\right\} : x \left\{\begin{matrix}\text{Wt. of}\\ MnO_2 \text{ present.}\end{matrix}\right.$$

The action is explained by the equation;

$$2FeSO_4+2H_2SO_4+MnO_2=Fe_2(SO_4)_3+MnSO_4+2H_2O.$$

SECTION X.

Quantitative Analysis of Magnesian Limestone.

594 Weigh roughly (551) 100 grains (or 6·5 grammes) of the mineral. Powder it finely (526), and preserve it in a corked tube.

595 *Estimation of the hygroscopic moisture.*—Weigh accurately (553) 50 grains (or 3 grammes), place it in a porcelain crucible, and heat it, in the air-bath, at about 300° F. (or 150° C.), for half an hour. Cover the crucible, allow it to cool in the desiccator (fig. 81) and weigh it. Repeat the weighing, at intervals of a quarter or half an hour, till the weight is constant. Calculate the percentage of water lost.

596 *Estimation of sand and clay.*—Weigh accurately (553) 25 grains (or 1·5 gramme) of the limestone, and treat it with hydrochloric acid as in (565), as long as anything more appears to be dissolved. Collect the undissolved residue (sand and clay) upon a filter (of about 3 inches diameter), taking care to avoid loss, and wash it with hot water (fig. 74), as long as a drop of the washings reddens blue litmus paper. Reserve the solution and washings for subsequent examination (597). Dry the residue upon the filter (540), incinerate (542) in a porcelain or platinum crucible, and weigh.

597 *Estimation of the silica* (contained in the limestone as a silicate).—Pour the solution from (596) or a part of it, down a glass rod into a half-pint evaporating dish, which must not be filled higher than within half an inch of the rim, and evaporate over a plain burner (fig. 22), without boiling; add the rest of the solution when the first has partly evaporated, and rinse out the beaker twice with a little water, into the dish. Continue to evaporate the liquid to dryness, placing a dial-

glass (convexity downwards) over the dish towards the end of the evaporation, to prevent loss from spurting. When the dish has cooled, pour about an ounce of water over the residue, and add a little hydrochloric acid, by degrees, applying a gentle heat, until either the residue has entirely dissolved, or nothing is left but white flakes of silica.* Add a few drops of nitric acid (to convert the ferrous chloride into ferric chloride) and heat gently for a minute or two. Dilute the solution with about two ounces of water, collect the silica upon a filter (about 3 inches in diameter), pouring the solution down a glass rod on to the filter, wash the silica with hot water till the washings (which must be saved) no longer redden blue litmus, dry (fig. 76), incinerate† very carefully in a porcelain or platinum crucible, and weigh.

598 *Estimation of the oxide of iron* (*ferrous oxide*) *and alumina.*—To the solution filtered from the silica in (597) add about 15 grains (or 1 gramme) of chloride of ammonium dissolved in water, and ammonia, until the solution smells strongly of it, even after stirring; heat the solution gently, in a beaker covered with a dial-glass, collect the precipitate (alumina and peroxide of iron) upon a filter (about 3 inches in diameter), wash with hot water till the washings (which must be saved) give no turbidity with oxalate of ammonia, dry (fig. 76), incinerate in a porcelain or platinum crucible, and weigh.

If the brown colour of the precipitate indicates the presence of any considerable quantity of iron, dissolve it in the crucible with a little hydrochloric acid, reduce the ferric chloride as in (586) and estimate the iron either by the standard solution of permanganate (583) or by the bichromate (590). From the weight of iron found, calculate the weight of peroxide of iron in the precipitate (56 parts of iron yield 80 parts of the peroxide), and deduct it from the joint weights of the alumina and peroxide of iron, to ascertain the amount of alumina. Since the iron is generally present in the limestone as a carbonate of the protoxide (ferrous carbonate), the weight of the protoxide corresponding to the metallic iron found must be calculated (7 parts of iron yield 9 parts of protoxide), and its percentage recorded.

599 *Estimation of the lime.*—To the solution and washings from the alumina and peroxide of iron in (598), add oxalate of ammonia in quantity more than

* Many limestones are free, or nearly so, from combined silica, and will therefore leave no residue undissolved at this stage.

† Silica is very apt, when heated, to be carried away by a current of air; it is therefore advisable to remove it as completely as possible from the filter, and to ignite it in a covered crucible, the filter being incinerated separately, and the ash added to the ignited silica after cooling.

sufficient to precipitate the whole of the lime;* heat the solution nearly to boiling in a beaker covered with a dial-glass, set it aside for some time, and proceed further as directed in (565), saving the filtered liquid and washings for the estimation of the magnesia.

600 *Estimation of the magnesia.*—Place the open beaker containing the filtered liquid and washings from (599) upon a piece of gauze over a lamp, and evaporate the liquid, without boiling it, till it measures only about four ounces; transfer it to a smaller beaker, rinsing the first with a little water; add ammonia in considerable excess, and precipitate the magnesia with phosphate of soda, proceeding as directed in (568).

601 *Estimation of the carbonic acid.*—Proceed according to the directions in (566).

602 Calculate the amount of each constituent in 100 parts of the limestone.—If the analysis be satisfactory, the sum of the percentages should amount to at least 99. The following statements will enable the analyst to corroborate his results or to discover which estimation is incorrect:—

If 44 parts of carbonic acid be allowed for every 56 parts of lime, and the same amount for every 40 parts of magnesia (and for every 72 parts of protoxide of iron if present) the sum of the amounts of carbonic acid thus calculated should be nearly the same as that obtained in the analysis.

If ten grains (or 0·6 gramme) of the powdered limestone be strongly heated in a porcelain or platinum crucible until its weight is constant, the total loss should, when calculated upon 100 parts, be equal to the joint percentages of carbonic acid and hygroscopic moisture.†

SECTION XI.

Quantitative Analysis of Copper Pyrites.

603 Weigh roughly 30 grains (or 2 grammes) of the mineral, and reduce it to a fine powder (526). Weigh accurately 25 grains (or 1·5 gramme), introduce it into a four-ounce beaker covered with a dial-glass (or into a flask with a small funnel, fig. 70); pour upon it, carefully (565), about three drachms (or 200 grain measures, or 10 cubic centimetres) of strong nitric acid; warm it gradually (fig. 68) until the action has in great measure

* 30 grains (or 2 grammes) dissolved in water will be sufficient for the purpose.

† Unless much iron be present, when a correction must be made for the absorption of oxygen to the amount of one-ninth of the weight of the ferrous oxide present.

ceased, add half an ounce (or 300 grain measures, or 15 cubic centimetres) of strong hydrochloric acid; heat to boiling, in the covered beaker, until the pyrites is completely acted on, and any separated sulphur has a yellow colour. Uncover the beaker, rinse the dial-glass with a few drops of water, and evaporate, without boiling, in the open beaker, till only enough liquid is left to cover the bottom. Should red fumes be still visible, add a little more hydrochloric acid, and again evaporate.*

604 *Estimation of the silica and unoxidized sulphur.*—Weigh a dried filter (562), about three inches in diameter; dilute the solution from (603) with two ounces (or 50 cubic centimetres) of water, and pour it through the filter, into a measure-flask (573) capable of containing 5000 grains (or 250 cubic centimetres). Collect every particle of the residue upon the filter, wash it till the washings are no longer acid, and dry it in the water-oven, till its weight is constant (562); deduct the weight of the filter, to obtain the joint weights of the silica and sulphur. Transfer the contents of the filter to a weighed porcelain crucible, incinerate the filter (542), collecting its ash in the same crucible, and heat the latter, until all the sulphur is consumed; weigh the residual silica and deduct its weight from the joint weights of silica and sulphur. Calculate the quantity of each in 100 parts of the mineral.

605 *Estimation of the sulphur which has been oxidized into sulphuric acid.*—Make up the volume of the filtrate and washings in the measure-flask (604) to 5000 grains (or 250 cubic centimetres) by the addition of water, taking great care to secure perfect mixture, which may be effected by adding the water with a pipette (fig. 91) thrust down to the bottom of the flask, and occasionally agitating. Take 2500 grains (or 125 cubic centimetres, or any other convenient fraction, if not too small) of the diluted solution, introduce it into a half-pint beaker, and proceed to estimate the sulphuric acid by precipitating with chloride of barium,† as in (556).

* A quicker process for dissolving the pyrites consists in treating it (in a dish) with HNO_3 and $KClO_3$. Add, to 0·5 grm. of pyrites, 25 c. c. HNO_3 (Sp. Gr. 1·4) and powdered $KClO_3$ amounting to about 3 times the bulk of the pyrites.

† About 30 grains of crystallized chloride of barium dissolved in water will be sufficient.

Calculate the amount of sulphur from the weight of the sulphate of baryta by the proportion :—

$$\begin{matrix} BaSO_4 \\ 233 \end{matrix} : \begin{matrix} S \\ 32 \end{matrix} :: \left\{ \begin{matrix} \text{Wt. of sul-} \\ \text{phate of} \\ \text{baryta} \\ \text{obtained.} \end{matrix} \right\} : x \left\{ \begin{matrix} \text{Wt. of sulphur} \\ \text{oxidized.} \end{matrix} \right.$$

Calculate the weight of sulphur oxidized upon 100 parts of the copper pyrites (recollecting that only half the solution prepared from the 25 grains has been employed), and add it to the percentage of unoxidized sulphur found in (604).

606 *Estimation of the copper.*—Transfer the other half of the solution taken for estimating the sulphuric acid in (605) to a pint beaker, dilute to about half a pint with water, and thoroughly saturate with hydrosulphuric acid gas (570). Allow the precipitate to subside, so that a little saturated solution of hydrosulphuric acid may be added to the liquid, to ascertain that all the copper is precipitated. Collect the sulphide of copper upon a filter (about 5 inches in diameter), wash it rapidly with water containing hydrosulphuric acid, until a drop of the washings is no longer tinged black by ammonia. Treat the filtrate and washings according to (607). Place the funnel containing the precipitated sulphide of copper over a half-pint flask, perforate the apex of the filter with a pointed glass rod (fig. 26), and wash as much as possible of the precipitate into the flask by means of a fine stream of water from a washing-bottle.* Dry the filter, incinerate it, and add the ashes to the contents of the flask; add to these two drachms (7 cubic centimetres) of nitric acid, and heat, at first gently, but finally to boiling, until the sulphur separates in yellow globules. Dilute the solution with two ounces (60 cubic centimetres) of water, filter, washing the filter thoroughly, and determine the copper as oxide, according to the directions given at (558). From the weight of the oxide, calculate that of the copper thus :—

$$\begin{matrix} CuO \\ 79{\cdot}5 \end{matrix} : \begin{matrix} Cu \\ 63{\cdot}5 \end{matrix} :: \left\{ \begin{matrix} \text{Wt. of oxide} \\ \text{of copper} \\ \text{obtained.} \end{matrix} \right\} : x \left\{ \begin{matrix} \text{Wt. of copper} \\ \text{found.} \end{matrix} \right.$$

* Great care must be taken to avoid washing any of the paper into the flask, since the presence of organic matter in the solution will prevent the complete precipitation of the copper by potash.

Calculate the copper in 100 parts of the pyrites (recollecting that only one-half of the solution from 25 grains has been employed).

607 *Estimation of the iron.*—Test the filtrate and washings from the sulphide of copper in (606) with hydrosulphuric acid, to ascertain that all the copper has been precipitated, and evaporate, in the beaker, to about a quarter of a pint. (A slight yellow precipitate of As_2S_3 is sometimes deposited.) Cover the beaker with a dial-glass, add a few drops of nitric acid, by degrees, continuing the application of heat, until all the iron is converted into peroxide, the liquid having a bright yellow colour. Add ammonia, by degrees, in slight excess; collect the precipitated peroxide of iron upon a filter (about 5 inches in diameter), wash with boiling water till the washings are no longer rendered turbid by nitric acid and nitrate of silver, dry (540), ignite (542) in a porcelain or platinum crucible, and weigh.

Calculate the weight of iron in the precipitate, thus:—

$$\underset{160}{Fe_2O_3} : \underset{112}{Fe_2} :: \left\{\begin{array}{c}\text{Wt. of per-}\\\text{oxide}\\\text{obtained.}\end{array}\right\} : x \left\{\begin{array}{c}\text{Wt. of iron}\\\text{found.}\end{array}\right.$$

Calculate the iron in 100 parts of the pyrites (recollecting that only one-half of the solution from 25 grains has been employed).

The sum of the percentages of copper, iron, sulphur, and silica should amount to at least 99, if the analysis be correct.

Calculate the formula of the mineral from its percentage composition thus: divide the percentage of each element by its atomic weight, and find the simplest ratio between the quotients. For example; a specimen contained 33 per cent. of Cu, 32 per cent. of Fe, and 33 per cent. of S.

$$\frac{33}{63{\cdot}5} = {\cdot}52 \text{ Cu}; \quad \frac{32}{56} = {\cdot}57 \text{ Fe}; \quad \frac{33}{32} = 1{\cdot}03 \text{ S}.$$

Whence the formula $CuFeS_2$ would be deduced.

SECTION XII.

Quantitative Analysis of Gunpowder; consisting of Nitre, Charcoal, and Sulphur.

608 *Estimation of the hygroscopic moisture.*—Weigh a porcelain crucible with its cover, and mark the

weight upon it, in ink (554). Introduce into it 100 grains (or 6 grammes) of gunpowder, and expose it in the water-oven (527) for an hour. Allow it to cool, in the covered crucible, and weigh it. Replace the crucible in the oven, and weigh it at intervals of a quarter of an hour till the weight remains constant.*

Fig. 95.

609 *Estimation of the nitre.*—Weigh a thin dry flask capable of holding four fluid ounces (fig. 95). Mark the weight upon the neck with a diamond or hard file. Attach it to a funnel by means of a perforated cork, fitted, air-tight, to the flask. Place in the funnel a filter which has been dried at 212° F. (562), and weighed. Moisten the filter, and fit it exactly to the sides of the funnel. Place upon it 100 grains (or 6 grammes) of gunpowder, and pour over this about half an ounce of boiling distilled water. Cover the funnel with a dial-glass, and allow it to stand for five minutes in order that the water may soak well into the gunpowder and dissolve the nitre. Loosen the cork to allow the air to escape from the flask, and the solution to pass through. Repeat this treatment with successive portions of boiling water, till all the nitre has been dissolved out, which may be ascertained by evaporating a drop from the funnel upon a slip of glass, when it should leave no residue of any consequence.†

If the funnel be fitted quite air-tight into the flask, the whole of the nitre may be very quickly extracted by this method. But the nitre may also be extracted by heating the gunpowder with two ounces of water in a beaker (fig. 68) for 15 minutes, allowing it to subside, and filtering (fig. 73) through the (weighed) filter, taking care to rinse every particle of the residue on to the filter with boiling water (536).

Proceed with the charcoal and sulphur, as in (610). Select a thin, shallow evaporating dish (*a*, fig. 96), and grease *very slightly* with a little lard, the upper margin of the inside of the dish, to the depth of an eighth of an inch, to prevent the nitre from creeping up the side of the dish. Weigh the dish and mark the weight upon it. Pour the solution of nitre into it, down a glass rod, till it

* Since a minute quantity of sulphur escapes at 212°, the moisture will be estimated a little too high. An exact determination would be made by drying at the ordinary temperature over oil of vitriol (546).

† About four ounces of water are usually required for this purpose.

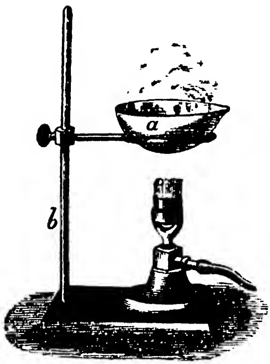


FIG. 96. Evaporation of the Solution of Nitre.

reaches the lower edge of the greased portion. Support the dish upon a retort-stand *b*, at some distance above a ring gas-burner, and evaporate the solution, without allowing it to boil, pouring in the remainder by degrees from the flask, and finally rinsing out the latter with water.* When the solution has been evaporated to about half an ounce, place the dish over a steam-bath (fig. 79), and continue the evaporation to perfect dryness. After thorough drying over the steam-bath, cover the dish with a dial-glass, and heat it gradually over a ring-burner (fig. 96), until the nitre begins to fuse. Allow the dish to cool, and weigh it.

610 *Estimation of the charcoal.*—Remove the filter containing the charcoal and sulphur (609) from the funnel, place it in a dish without unfolding it, and dry it in the water-oven (fig. 67) for an hour. Weigh it *rapidly* to avoid absorption of moisture from the air; replace it in the water-oven, and weigh it at intervals of half an hour till its weight is constant. Place the filter upon the same funnel (which, as well as the flask, should have been thoroughly dried), and spirt upon it, from a small washing-bottle, about two drachms of pure bisulphide of carbon.† Cover the funnel, and allow it to remain for five minutes, Loosen the cork from the flask, so that the bisulphide may run through, and repeat the washing several times until the whole of the sulphur is dissolved out, which may be ascertained by evaporating a few drops as they fall from the funnel, when they should leave no residue. Proceed with the solution of sulphur

* Should any charcoal have passed through the filter, it should be now collected upon a small dried and weighed filter, well washed, its weight determined and added to that obtained in (610).

† Since the bisulphide of carbon is very volatile and inflammable, great care must be taken not to bring it near a flame. Its purity must be tested by allowing a little to evaporate in a watch-glass, when it should leave no residue.

in bisulphide of carbon according to (611). Place the filter with the charcoal in a dish, and allow it to remain exposed to the air until the bisulphide of carbon has evaporated, which may be known by the smell. Then place it in the water-oven, and weigh it at intervals until its weight is constant. On deducting the weight of the filter (609), that of the charcoal will be ascertained.

611 *Estimation of the sulphur.*—To the flask containing the solution of sulphur in bisulphide of carbon, fit a cork and tube bent as in the figure (fig. 97),

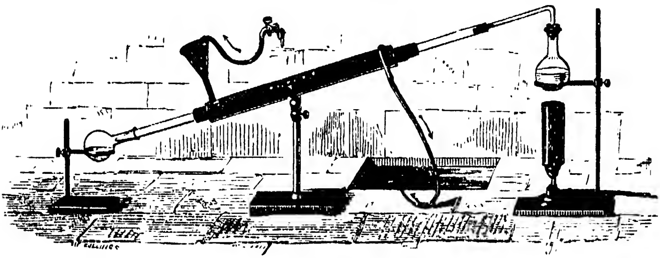


Fig. 97. Distillation of the Bisulphide of Carbon.

and connected with a Liebig's condenser. Apply a very gentle heat with a ring burner, and distil off the greater portion of the bisulphide of carbon. When only a shallow layer of the latter remains at the bottom of the flask, disconnect it, and evaporate the remainder by gently blowing into the flask with a moderately wide tube. When the odour of bisulphide of carbon has entirely disappeared, place the flask in a water-oven, and weigh it at intervals as long as any variation in weight is perceptible. The weight of the sulphur is then ascertained by deducting that of the flask.

612 To corroborate the above result, the sulphur may be estimated in another portion of the gunpowder by converting it into sulphuric acid, and weighing this in the form of sulphate of baryta. For this purpose, 50 grains (or 3 grammes) of gunpowder are heated in a small flask, with one fluid ounce of concentrated nitric acid (S.G. 1·36), and crystals of chlorate of potash are added in small portions to the hot liquid until the whole of the powder is

dissolved to a clear light yellow liquid. This will require about 50 grains (3 grammes) of the chlorate.* The solution is boiled down, rinsed into a beaker, diluted with at least six ounces of hot water (to avoid precipitation of nitrate of baryta), and mixed with about 50 grains of chloride of barium dissolved in an ounce of hot water. The precipitate of sulphate of baryta is allowed to subside, collected on a filter, washed with the precautions mentioned in (557), and the determination completed as therein directed.

The percentage of sulphur found by this process is usually somewhat higher than that obtained by treatment with bisulphide of carbon, which fails to extract the last traces of sulphur from the charcoal. Should the difference be only 0·1 or 0·2 per cent., the mean may be taken as correct.

613 If the analysis is correct, the sum of the charcoal, sulphur, and nitre should amount to at least 99·5. The sum of the charcoal and sulphur, separately determined in (610) and (611), should be nearly equal to their joint weight determined in (610).

* The complete oxidation of the charcoal sometimes occupies several hours, and may require even more potassium chlorate than that prescribed.

SECTION XIII.

Additional Exercises in Quantitative Analysis.

614 Proficiency in quantitative analysis can only be attained by much practice. The directions already given will enable the student to perform several other analyses. The following examples may be recommended:—

Sulphate of Iron (green vitriol); estimate the sulphuric acid by (556), and the oxide of iron by (583, 590, or 607).

Chloride of Barium; estimate the chlorine by (563), and the barium by precipitation with sulphuric acid (556).

Carbonate of Magnesia; estimate the magnesia by (568), and the carbonic acid by (566).

Phosphate of Soda; estimate the phosphoric acid by precipitating with a mixture of sulphate of magnesia with chloride of ammonium and ammonia, and proceeding as in (568).

Iron Pyrites; treat as for copper pyrites, omitting the estimation of copper.

PART V.

REAGENTS.

CHAPTER I.

EXAMINATION AND USES OF REAGENTS.

615. The following is a list of the reagents, &c., usually employed in testing and analysis, with the quantities in which it is generally necessary to provide them :—

Solutions in narrow-mouthed 8-oz. bottles.

1. Sulphuric acid, strong
2. „ „ dilute
3. Hydrochloric „ strong
4. „ „ dilute
5. Nitric „ strong
6. „ „ dilute
7. Hydrosulphuric acid
8. Ammonia
9. Carbonate of ammonia
10. Chloride of ammonium
11. Potash
12. Baryta-water

Solutions in narrow-mouthed 4-oz. bottles.

1. Acetic acid
2. Tartaric „
3. Hydrosulphate of ammonia
4. Lime-water
5. Sulphate of lime
6. Chloride of calcium
7. Chloride of barium
8. Nitrate of silver
9. Alcohol

Solutions in narrow-mouthed 2-oz. bottles.

1. Oxalate of ammonia
2. Ferrocyanide of potassium
3. Ferridcyanide „
4. Antimoniate of potash
5. Carbonate of soda
6. Phosphate „
7. Nitrate of baryta
8. Sulphate of magnesia
9. Perchloride of iron
10. Nitrate of cobalt
11. Sulphate of copper
12. Acetate of lead
13. Perchloride of mercury
14. Protochloride of tin
15. Bichloride of platinum
16. Solution of starch

Solids in wide-mouthed 2-oz. bottles.

1. Phosphate of soda and ammonia, 1 oz.
2. Nitrate of potash, 2 oz.
3. Cyanide of potassium, 1 oz.
4. Borax, 1 oz.
5. Sulphate of iron, 1 oz.
6. Flowers of sulphur, 1 oz.
7. Carbonate of soda, 2 oz.
8. Nitrate of baryta, 2 oz.

In addition to these, the analyst will require—

Granulated zinc, 1 lb.
Blue and red litmus, and turmeric paper.
Distilled water.

Stoppered bottles are to be preferred for the reagents. Those made of green glass are the least expensive and the least corroded.

The following reagents will eventually be required, but they are not indispensable at the commencement :—

Solutions.

1. Oxalic acid, 2 oz.
2. Hydrofluosilicic acid, 2 oz.
3. Carbonate of potash, 2 oz.
4. Iodide of potassium, 2 oz.
5. Chromate of potash, 2 oz.
6. Acetate of potash, 2 oz.
7. Sulphate of manganese, 2 oz.
8. Sulphindigotic acid, 2 oz.

The beginner should also be provided with the following articles :—

12 test-tubes.
Rack with draining-pegs.
Tube-cleaner.
Spirit-lamp or gas-burner.
3 funnels : 2 oz., 1 oz., ½ oz.
Filter-paper.
Slips of window-glass.
Glass rod.
Narrow hard glass tubing.
Blowpipe.
Triangular file.
Platinum wire and foil.
4-oz. evaporating-dish.
Half-pint Wedgwood mortar.
Wire triangle.
Wire tripod, or retort stand.

616. Most of the chemicals, as they are met with in commerce, being always more or less impure, and, as those even which are sold in the shops as pure reagents, are not unfrequently found, on examination, to be otherwise ; it is always necessary, before taking a reagent into use, to ascertain by experiment whether it is of sufficient purity for the purposes for which it is intended. It may be stated as a general rule, that, when a chemical substance is required for use in analysis, it ought to be as nearly pure as possible : while, for many of the other operations of chemistry, the substances which are usually met with in commerce are sufficiently pure. The following brief remarks relative to the more common impurities of reagents, together with their principal uses, will probably be found useful to the student.

The substances are arranged alphabetically for more convenient reference.

617. *Acetate of Lead* (*Plumbic Acetate*), $Pb(C_2H_3O_2)_2$.—Acetate of lead is used as a test for several acids which form with oxide of lead insoluble salts. For testing, one part of the salt may be dissolved in ten parts of water.

618. *Acetate of Potash* (*Potassium Acetate*), $KC_2H_3O_2$.—The commercial salt is liable to contain carbonate of potash, which causes it to effervesce when mixed with hydrochloric acid. Solution of acetate of potash should not be tinged by hydrosulphate of ammonia. The salt may be dissolved for use in three times its weight of water.

619. *Acetic Acid*, $HC_2H_3O_2$.—This acid is often contaminated with one or more of the following substances: sulphuric, sulphurous, and hydrochloric acids, lead, and certain saline matters.

(*a*) Any fixed impurity may be detected by heating a little on a slip of glass.

(*b*) Add to a portion of the diluted acid, a solution of chloride of barium: if sulphuric acid is present, a white precipitate is thrown down.

(*c*) Test for sulphurous acid by (372).

(*d*) Nitrate of silver, added to the diluted acid, gives a white curdy precipitate, which is insoluble in nitric acid, if any hydrochloric acid is present.

(*e*) Neutralize a small portion with ammonia, and add hydrosulphuric acid or hydrosulphate of ammonia: if lead or any other metallic substance is present (except the alkalies and alkaline earths), a precipitate is produced.

Acetic acid is chiefly employed in the laboratory as a solvent, and for the purpose of acidifying solutions, in cases where hydrochloric and nitric acids would act prejudicially.

620. *Alcohol*, C_2H_6O.—The alcohol commonly used in chemical experiments should have a specific gravity of about 0·83, except in cases where absolute alcohol is required, when it should be 0·796.* When evaporated on glass, it should leave no residue, and should not change the colour of litmus paper. It is used chiefly as a solvent, and for the purpose of facilitating the precipitation of substances which are less soluble in it than in water.

621. *Ammonia*, NH_3.—The liquid ammonia of the shops is generally sufficiently pure for most purposes of analysis; it sometimes, however, contains traces of carbonate, sulphate, and muriate of ammonia, and occasionally chloride of calcium. The carbonate is detected by adding lime water; the sulphate by supersaturating with dilute hydro-

* The strength of any sample may be inferred from its specific gravity by reference to Table VII. in the Appendix.

chloric acid, and testing with chloride of barium; the chloride of ammonium may be detected by supersaturating with nitric acid, and adding nitrate of silver; and the lime (chloride of calcium) with oxalate of ammonia.

Ammonia is used chiefly for the purpose of neutralizing acid solutions, and for precipitating metallic oxides from their solutions, most of which are decomposed by it.

Its specific gravity should be about 0·88 (see Table VI. in Appendix). For ordinary use, it is advisable to dilute it with an equal bulk of water.

622. *Antimoniate of Potash* (*Potassium Antimoniate*), $K_2H_2Sb_2O_7$.—This substance seldom or never contains any impurity that can interfere with its action as a test for soda, which is the only use to which it is applied in the laboratory. One part of the ordinary antimoniate should be boiled with twenty parts of water and filtered. The solution must be kept in a well-stoppered bottle, and carefully excluded from the air, as the carbonic acid is liable to decompose it, and cause a precipitation of antimonic acid. Its solution should give a decided crystalline precipitate, with even a dilute solution of carbonate of soda.

623. *Baryta Water*, BaH_2O_2.—In solution, hydrate of baryta is employed in order to separate magnesia from the alkalies; it is prepared by boiling the crystallized hydrate with about 20 parts of water. The solution must be preserved from contact with the air, for the carbonic acid of the latter will precipitate the baryta as carbonate.

The dry hydrate is used in the decomposition of insoluble silicates in order to test them for alkalies; it should therefore be free from these latter, which may be ascertained by dissolving in dilute hydrochloric acid, precipitating the baryta by ammonia and carbonate of ammonia, with the aid of heat, filtering, evaporating to dryness, and igniting, when no residue should be left.

624. *Bichloride of Platinum* (*Platinic Chloride*), $PtCl_4$.—Bichloride of platinum is employed only as a test for potash and ammonia; it is prepared by dissolving scraps of platinum foil (previously cleaned by boiling in strong hydrochloric acid) in nitro-hydrochloric acid, and evaporating this solution at a moderate heat to a syrupy consistence; it may be dissolved in so much water that the solution gives an immediate crystalline precipitate with a

moderately dilute solution of chloride of potassium or chloride of ammonium.

625. *Black Flux.*—Black flux is an intimate mixture of carbonate of potash and finely divided charcoal, and is prepared by deflagrating in an iron spoon or crucible a mixture of two parts of bitartrate of potash and one of nitre. It is used as a reducing flux in blowpipe experiments. For most purposes, an intimate mixture of eight parts of dry carbonate of soda and one part of very finely powdered wood charcoal will answer equally well.

626. *Borax* (*Biborate of Soda*) (*Acid Sodium Borate*), $Na_2O.2B_2O_3$.—Borax occasionally contains traces of sulphate and chloride, which may be detected in the same way as in the phosphate of soda (655). It is employed almost exclusively as a flux in blowpipe experiments, for which purpose it is admirably adapted; the second equivalent of boracic acid which it contains, exerts a strong attraction for bases at a high temperature, and is capable of displacing several acids from their combinations; it also forms many double compounds and mixtures which are readily fusible.

627. *Carbonate of Ammonia* (*Ammonium Carbonate*), $(NH_4)_2CO_3$.—The common carbonate of ammonia is a sesquicarbonate, or a compound of the neutral carbonate and the bicarbonate. When the neutral carbonate is required, and it is the best suited for most purposes of analysis, it may be prepared in solution by dissolving one part, by weight, of the sesquicarbonate, in three or four parts of water, and adding one part of liquid ammonia (sp. gr. 0·96). It is frequently employed in analysis, to precipitate some of the metals as carbonates; it is also used to neutralize acid solutions, and for other purposes.*

628. It is occasionally contaminated with traces of animal oil, chloride of ammonium, sulphate of ammonia, and oxide of iron.

(*a*) Heat a small fragment on platinum foil; if any fixed saline impurity is present, it will be left after ignition, and if any charring takes place, it indicates the presence of animal matter.

(*b*) Supersaturate a little of the solution with nitric

* A saturated solution of the sesquicarbonate, prepared by agitating an excess of the salt with cold water (about 1 part of the salt to 3 of water), is required for some purposes, as for the separation of tin and antimony.

acid, and add to one portion a few drops of chloride of barium; a white precipitate, insoluble in nitric acid, indicates sulphuric acid.

(*c*) To the other portion of the acid solution, add nitrate of silver; if any chloride of ammonium is present, it will cause a white curdy precipitate.

(*d*) Add a little hydrosulphate of ammonia to the solution; a dark tinge indicates the presence of a trace of iron.

629. *Carbonate of Potash* (*Potassium Carbonate*), K_2CO_3.—This salt generally contains traces of sulphate and chloride, and occasionally silica and alumina.

(*a*) A solution, supersaturated with hydrochloric acid, and tested with chloride of barium, gives a white precipitate if any sulphuric acid is present.

(*b*) A solution acidified with nitric acid gives, with nitrate of silver, a white curdy precipitate, if it contains chloride of potassium.

(*c*) Neutralize a portion of the solution with hydrochloric acid, and evaporate to dryness; if the residue does not wholly dissolve when treated with hydrochloric acid, silica is probably present.

(*d*) If ammonia causes, in the hydrochloric solution, a white gelatinous precipitate, alumina is probably present.

Carbonate of potash is frequently employed to precipitate metallic oxides and carbonates from their soluble combinations, and for the purpose of neutralizing acid solutions.

630. *Carbonate of Soda* (*Sodium Carbonate*), Na_2CO_3.—The best method of preparing pure carbonate of soda is to ignite the bicarbonate, when the second equivalent of carbonic acid and the water are expelled, and pure anhydrous carbonate is left. The salt of commerce frequently contains a little sulphate and chloride, which may be detected in the manner already detailed (629, *a* and *b*). The more impure varieties contain also traces of sulphide of sodium, and sulphite and hyposulphite of soda. If sulphide of sodium be present, hydrosulphuric acid (recognised by paper moistened with acetate of lead) will be evolved on adding hydrochloric acid in excess. If sulphite of soda be present, the acid solution will smell of sulphurous acid, and will evolve hydrosulphuric acid when a fragment of zinc is dropped into it. The pre-

sence of hyposulphite of soda would cause the acid solution to become turbid when heated, at the same time disengaging sulphurous acid.

It is employed for the same purposes as carbonate of potash (629), also as a flux for the blowpipe, and for fusing with insoluble silicates,* &c. For use as a liquid reagent, one part of the crystallized salt may be dissolved in three parts of water.

631. *Chloride of Ammonium* (*Ammonium Chloride*), NH_4Cl.—The sal-ammoniac of commerce usually contains a considerable quantity of iron; and must therefore be purified, for analytical purposes, by crystallization.† If pure, it should be entirely volatilized when heated on platinum foil, and its solution should not give any precipitate with chloride of barium or dark tinge with hydrosulphate of ammonia. One part of the salt should be dissolved in eight parts of water.

632. *Chloride of Barium* (*Barium Chloride*), $BaCl_2$.—Chloride of barium sometimes contains traces of iron and lime. It should not be discoloured by hydrosulphate of ammonia, and, after being treated with a slight excess of sulphuric acid, and filtered, the clear solution should leave no fixed residue when evaporated on platinum foil; because the whole of the baryta is separated by the sulphuric acid, and any other fixed matter must be some impurity.

It is used chiefly for the purpose of testing for acids, especially sulphuric, with which it forms the insoluble sulphate of baryta. For use, one part of the salt may be dissolved in ten parts of water.

633. *Chloride of Calcium* (*Calcium Chloride*), $CaCl_2$.—This substance occasionally contains a little free lime or hydrochloric acid, and traces of iron. The first are

* The *carbonate of potash and soda*, prepared by incinerating Rochelle salt (tartrate of potash and soda) is generally used for the decomposition of silicates, being much more fusible than the simple carbonates. A mixture of 53 parts of dry carbonate of soda with 69 parts of dry carbonate of potash will answer the purpose.

† Dissolve the sal-ammoniac of commerce in about three parts of hot water; add to the solution a little ammonia and some freshly prepared hydrosulphate of ammonia as long as it produces a darker tinge in the liquid. Set it aside for twelve hours, in order that the sulphide of iron may be deposited, filter and evaporate the clear solution till it begins to crystallize, when it may be set aside. The product may be purified by again dissolving in water and recrystallizing.

detected by test papers, and the last, if present, causes hydrosulphate of ammonia to throw down in the solution a black precipitate, or to impart a greenish tint to the liquid. As a reagent, chloride of calcium is employed chiefly in testing for some of the organic acids. It is also of great use in the laboratory as a drying agent, having so strong an attraction for water, that a moist gas passed over it is rapidly and completely deprived of its water. For this purpose the chloride need not be absolutely pure; it should not be fused, but merely dried, as the unfused is more porous, and consequently offers a larger amount of surface to any gas passed over it. A solution of crystallized chloride of calcium in three parts of water may be used as a reagent.

634. *Chromate of Potash* (*Potassium Chromate*), K_2CrO_4. —This salt occasionally contains traces of sulphate of potash, which is readily detected by precipitating a little of the solution with chloride of barium, and adding an excess of hydrochloric acid which redissolves the chromate of baryta, while any sulphate remains insoluble.

It is employed as a test for several of the metallic oxides, with many of which it forms insoluble salts (chromates) of characteristic colours, as the chromate of lead, which is bright yellow. For use as a reagent it may be dissolved in ten times its weight of water.*

635. *Cyanide of Potassium* (*Potassium Cyanide*), KCN. —Cyanide of potassium is sometimes used in blowpipe experiments, and also as a liquid test. It should be white, and entirely soluble in water.

636. *Ferricyanide of Potassium* (*Red Prussiate of Potash*), K_3Cy_6Fe.—It occasionally contains traces of the yellow prussiate, which is easily detected by the solution giving a blue precipitate or green colour with perchloride of iron (313). It is used as a test for the protosalts of iron, with which it forms a blue precipitate, which is similar in appearance to that formed by ferrocyanide of potassium with the persalts (312). It may be dissolved in ten or fifteen parts of water.

637. *Ferrocyanide of Potassium* (*Yellow Prussiate of Potash*), K_4Cy_6Fe.—This salt, as met with in commerce,

* The bichromate is a better salt to keep in solution, as the chromate attacks the glass of the bottles and renders it opaque.

is sufficiently pure for the purposes of testing. It is employed as a test for the persalts of iron, with which it forms a deep blue precipitate of Prussian blue. It gives characteristic precipitates, also, with some other metals. For use as a reagent, one part of the salt may be dissolved in fifteen or twenty parts of water.

638. *Hydrochloric Acid*, HCl.—This acid, in the form met with in commerce, is never pure, usually containing sulphurous acid, sulphuric acid, and chloride of iron, and occasionally free chlorine and chloride of arsenic.

(*a*) Evaporate a drop or two on a piece of glass: if pure, no residue is left.

(*b*) Dilute a portion with four or five times its bulk of distilled water, and add a drop of chloride of barium: if sulphuric acid is present, a white precipitate is produced.

(*c*) Add ammonia in excess, and a drop of hydrosulphate of ammonia: a black or green precipitate indicates iron.

(*d*) Add a little of the acid to water tinged with sulphate of indigo: if it contains free chlorine, the blue colour is bleached.

(*e*) Arsenic may be detected by Marsh's or Reinsch's test (257, 267).

(*f*) Sulphurous acid may be detected by dissolving a fragment of zinc in the acid, when hydrosulphuric acid will be evolved, and may be recognised by paper moistened with acetate of lead (372).

The uses of hydrochloric acid are very numerous, especially in analysis, in which it is of constant value as a solvent for substances which are insoluble in water; many of the metals dissolve readily in it, forming soluble chlorides, and it is occasionally used to precipitate silver and mercury from their solutions.

When *dilute* hydrochloric acid is required, the strong acid may be diluted with about twice its bulk of water. The strong acid should have the specific gravity 1·20 (see Table III. in Appendix).

639. *Hydrofluosilicic Acid*, $2HF.SiF_4$.—In order to prepare this reagent, a mixture of equal parts of finely powdered fluor spar and sand (or better, powdered glass) is heated with six parts of concentrated sulphuric acid in a large dry flask. The gaseous fluoride of silicon thus evolved, is conducted through a dry tube bent twice at

right angles, into a glass filled with water, but containing at the bottom a sufficient quantity of mercury to cover the orifice of the delivery tube, in order to prevent its being choked by the gelatinous silica which is deposited as soon as the gas meets the water (406). The solution is filtered from the silica and is ready for use. It should give a decided crystalline precipitate with chloride of barium, but none with the chlorides of calcium and strontium.

Hydrofluosilicic acid is chiefly employed as a test for baryta.

640. *Hydrosulphate of Ammonia* (*Sulphide of Ammonium*) $(NH_4)_2S$.—Hydrosulphate of ammonia is prepared by passing a stream of hydrosulphuric acid gas (641) through a solution of ammonia until it is saturated.* To ascertain whether the saturation is complete, a few drops may be tested with sulphate of magnesia; if the ammonia is saturated, this gives no precipitate; but if any free ammonia is left, it throws down the hydrate of magnesia. When first prepared, the solution is almost colourless, but it gradually becomes yellow, owing to partial decomposition, the oxygen of the air combining with the hydrogen, while sulphur is set free, and remains dissolved: when this decomposition has taken place, the addition of an acid causes not only the evolution of hydrosulphuric acid, but also precipitates the dissolved sulphur† (379).

Hydrosulphate of ammonia is much used, both in qualitative and quantitative analysis, chiefly for the purpose of precipitating certain metals from their solutions, and for separating the metals of the second class from the alkalies and alkaline earths.

641. *Hydrosulphuric Acid* (*Sulphuretted Hydrogen*) H_2S.—This reagent, whether required in the gaseous form or in solution, is always prepared in the laboratory. Fragments of sulphide (sulphuret) of iron are placed in a bottle (fig. 98), together with three or four ounces of water; a little strong sulphuric acid is then added, which disengages the gas, sulphate of iron being produced. The

* Strictly speaking, it is the *ammonium hydrosulphide* NH_4HS which is thus obtained. $NH_3+H_2S=NH_4HS$. This is sometimes mixed with an equal volume of NH_3 to furnish ammonium sulphide. $NH_4HS+NH_3+(NH_4)_2S$.

† Very yellow hydrosulphate of ammonia is unfit for use, on account of the ease with which it deposits sulphur, even on dilution. When too yellow for use, it gives a red (instead of black) precipitate with acetate of lead.

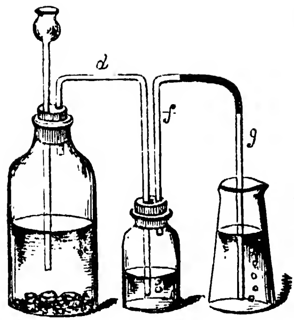


Fig. 98. Sulphuretted hydrogen Apparatus.

gas thus formed is passed through water contained in the second bottle, for the purpose of purifying it from any sulphuric acid and iron that may have been carried over mechanically, and is then conducted, by the bent tube, *f*, into a bottle of distilled water, when an aqueous solution of the gas is required, or into a glass containing any solution which it is intended to act upon.

It must be borne in mind when experimenting with this gas, that it is not only highly offensive, but poisonous, and induces headache and nausea even when largely diluted with air: on this account it should be prepared either near a ventilating flue, or in the open air, never in a close room.

In most cases of mere testing, the aqueous solution is the most convenient form in which to apply it. The water should be saturated with the gas, of which it is capable of retaining in solution about two or three times its volume; this may be judged of by its strong disagreeable smell, resembling that of rotten eggs, and by its giving a copious white precipitate of sulphur when treated with perchloride of iron (308). It should also be tested for iron, which it sometimes contains when carelessly prepared: if such is the case, it becomes dark-coloured on the addition of ammonia, owing to the formation of sulphide of iron. The solution should not be kept long, as it is liable to decompose, unless carefully closed from the air, the oxygen of which combines with the hydrogen to form water, while sulphur is deposited.*

When it is required to precipitate, by hydrosulphuric acid, *the whole* of any metal in a solution, it is often necessary to pass the gas at once into it; and this should be

* In order to keep it as long as possible, the stopper should be greased, and the bottle kept inverted. When it is decomposed, the same (and not fresh) water should be again saturated with the gas, since all the free oxygen dissolved in it has been consumed.

continued until the liquid is saturated, which is known by removing the gas-delivering tube, and blowing away the superincumbent air: when, if it smells distinctly of the gas, the solution may be considered saturated, and the whole of the metal must have been converted into sulphide.

The important uses to which hydrosulphuric acid is applied, render it of great value in many processes of analysis. It precipitates many of the metals from their solutions as insoluble sulphides; and is one of the reagents employed in determining the class to which an unknown metal in solution belongs. It is also extensively used in quantitative analysis, on account of the perfect manner in which it separates the whole of many of the metals from their solutions. Hydrosulphuric acid is also sometimes useful as a deoxidizing agent, reducing metallic oxides in solution to a lower degree of oxidation, as the peroxide of iron to the protoxide; this property is owing to the facility with which it is decomposed, yielding up its hydrogen to the oxygen of the oxide, while the sulphur is usually set free (308).

642. *Iodide of Potassium* (*Potassium Iodide*), KI.—Iodide of potassium is often adulterated with carbonate of potash; sulphate of potash and chloride of potassium are also often present. It should always be in the form of well-defined (cubical) crystals, as the adulterated varieties are readily distinguishable by their imperfect crystalline form.

(*a*) Add a little dilute hydrochloric acid: if effervescence takes place, some carbonate is present.

(*b*) If sulphates are present, they may be detected by adding chloride of barium, which will, in that case, cause a white precipitate, insoluble in hydrochloric acid.

(*c*) Add a little nitrate of silver: this will cause a pale yellow precipitate of iodide of silver, together with chloride of silver, in case any soluble chloride is present. To separate them, filter the mixture, and after washing the precipitate, treat it with a slight excess of ammonia, which dissolves the chloride, and leaves the iodide undissolved; on neutralizing the ammoniacal solution with nitric acid, the appearance of a white curdy precipitate indicates the presence of a chloride.

Iodide of potassium is employed chiefly as a test for

lead, mercury, and occasionally some of the other metals. For use as a reagent, one part of the salt may be dissolved in twenty parts of water.

643. *Lime Water*, CaH_2O_2.—This reagent is prepared by digesting hydrate of lime in cold distilled water for an hour or two, stirring the mixture occasionally, and when the undissolved portion of the lime has subsided, pouring off the clear solution. As it is liable to spoil when exposed to the air, owing to the absorption of carbonic acid, it should be kept in a well-stoppered bottle.

Lime water should be sufficiently strong to turn the yellow colour of turmeric instantly and decidedly brown: and, when tested with carbonate of soda, should throw down a copious white precipitate of carbonate of lime. It is used as a test for carbonic acid and some of the organic acids.

644. *Nitrate of Baryta* (*Barium Nitrate*), $Ba(NO_3)_2$.—Nitrate of baryta is liable to the same impurities as chloride of barium (632), and they may be detected in the same way. It should also be free from any chloride, which may be known by adding nitrate of silver. Its uses are the same as those of chloride of barium, for which it is occasionally substituted in cases when the addition of the chloride would interfere with the subsequent stages of an analysis, as when we have to test for chlorides in the same solution. For use, it may be dissolved in ten parts of water.

Nitrate of baryta is also employed in the examination of insoluble substances for alkalies. It should be tested for these in the same manner as hydrate of baryta (623).

645. *Nitrate of Cobalt*, $Co(NO_3)_2$.—This reagent is used chiefly for the detection of alumina, zinc, magnesia, and some other substances, by means of the blowpipe. The solution employed for this purpose may contain one part of the salt dissolved in ten of water. It should be kept in a small bottle, furnished with a cork and dropping-tube.

646. *Nitrate of Potash* (*Potassium Nitrate*), KNO_3.—Nitrate of potash often contains traces of sulphate and chloride, and occasionally nitrates of soda and lime.

(*a* and *b*) The sulphate and chloride may be detected with chloride of barium and nitrate of silver.

(*c*) If lime be present, it causes a precipitate when the solution is treated with oxalate of ammonia.

(*d*) The presence of nitrate of soda causes the salt to impart a yellow colour to flame.

It is used almost exclusively in the dry state, for the purpose of oxidizing substances which resist other methods of oxidation; this property is owing to the oxygen of the nitric acid being loosely combined, and at a high temperature readily yielded up to any substance which has a strong attraction for it, such as sulphides, organic matters, &c.

647. *Nitrate of Silver* (*Argentic Nitrate*), $AgNO_3$.—This reagent is sometimes adulterated with nitrate of potash, and occasionally contains traces of copper and lead. When precipitated by a slight excess of hydrochloric acid, the filtered solution ought to leave no fixed residue when evaporated on platinum foil, as the whole of the silver would be thrown down, and any impurity would remain in solution. Copper is detected by adding ammonia in excess to the solution, when it will give the liquid a blue tinge. Nitrate of silver is used chiefly as a test for chlorine (chlorides and hydrochloric acid), and also for phosphoric and some of the other acids. For use as a reagent, one part of the salt may be dissolved in twenty parts of water.

648. The *ammonio-nitrate of silver*, used as a test for arsenic, is prepared by adding ammonia to a solution of the nitrate, until the precipitate at first thrown down is just redissolved.

649. *Nitric Acid*, HNO_3.—Nitric acid, as met with in commerce, usually contains sulphuric and hydrochloric acids, and occasionally a little fixed saline matter. If it be red or yellow, it contains nitrous acid, which does not interfere with its ordinary uses in the course of analysis.*

(*a*) The saline matter may be detected by evaporating a few drops on a slip of glass, when any fixed impurities will be left.

Dilute a little of the acid with water, and divide it into two portions.

* Iodic acid is occasionally present in nitric acid, and may be detected in the residue after evaporating to dryness, by adding starch and a little sulphurous acid, which will give a blue colour.

(*b*) To the first, add chloride of barium: if a white precipitate is produced, sulphuric acid is present (87).

(*c*) To the other, add nitrate of silver: a white precipitate, soluble in ammonia, indicates hydrochloric acid.

Nitric acid is used chiefly as a solvent for substances which are insoluble in water, especially for some of the metals, which it readily oxidizes, and converts into nitrates, nearly all of which are soluble in water. It is also frequently employed to raise compounds to a higher state of oxidation, as in converting the protoxide of iron into the peroxide.

When *dilute* nitric acid is required, it may be prepared by mixing one part of the strong acid with two parts of distilled water. The specific gravity of the strong acid should be 1·42 (see Table II. in Appendix).

650. *Nitrohydrochloric Acid* (*Aqua Regia*).—This is always prepared when required, by mixing together strong nitric and hydrochloric acids, usually in the proportion of one part of nitric to four of hydrochloric. Its chief uses depend on its intense oxidizing or chlorinizing properties, whereby the most refractory metals, some of which resist the action of all other acids, are brought into solution.

651. *Oxalate of Ammonia* (*Ammonium Oxalate*), $(NH_4)_2C_2O_4$—This salt, as met with in the shops, is sufficiently pure for all purposes of analysis.* Like oxalic acid, it is employed chiefly for the purpose of precipitating lime from its solutions; for this purpose, it may be dissolved in about six times its weight of water.

652. *Oxalic Acid*, $H_2C_2O_4$.—Oxalic acid occasionally contains traces of nitric acid (which causes it to deliquesce in damp air, and to have a slightly acid smell), and also fixed saline matter.

(*a*) The former may be detected by sulphate of iron (367).

(*b*) The latter is left as a fixed residue after ignition on platinum foil.

It may be easily purified by recrystallization.

The chief use to which oxalic acid is applied in analysis,

* The oxalate at present found in commerce often changes after a short time into carbonate, when kept in solution. It then effervesces in addition of HCl.

is to precipitate lime from its solutions. For use as a test, one part of the crystallized acid may be dissolved in ten parts of water.

653. *Perchloride of Iron* (*Ferric Chloride*), Fe_2Cl_6.—This salt is liable to contain a little free acid, and traces of the protochloride. The free acid is detected by adding a very little ammonia, when the first precipitate will disappear on shaking, if free acid be present. If any protosalt of iron is present, the solution gives a blue colour with ferridcyanide of potassium. It is used as a test for some of the organic acids, and is also sometimes useful in the determination of phosphoric acid. It may be dissolved in five parts of water.*

654. *Perchloride of Mercury* (*Mercuric Chloride*), $HgCl_2$.—This is occasionally employed as a test for tin, for hydriodic and some other acids, and also for some kinds of organic matter: it may be dissolved in twenty parts of water.

655. *Phosphate of Soda* (*Hydro-di-sodic Phosphate*), Na_2HPO_4.—This salt sometimes contains a little sulphate and chloride. To detect these impurities, add to one portion, in solution, chloride of barium, and to the other nitrate of silver, and supersaturate both with nitric acid; if the precipitate does not entirely dissolve in either case, a sulphate or chloride is present.

It is employed chiefly as a test for magnesia, with which it forms, in the presence of ammoniacal salts, the double phosphate of magnesia and ammonia. For the purposes of testing, it may be dissolved in ten parts of water.

656. *Phosphate of Soda and Ammonia* (*Microcosmic Salt*).—This salt occasionally contains traces of chloride of sodium, which may readily be detected by adding a few drops of nitrate of silver to a solution of the salt, acidified with nitric acid, when a curdy white precipitate indicates the presence of the chloride.

Microcosmic salt is used almost exclusively in blowpipe experiments; when heated, it is decomposed, the ammonia and water are expelled, and metaphosphate of soda is left.

657. *Potash* (*Potassium Hydrate*), KHO.—On account of its strong attraction for many substances, caustic pot-

* It is usually purchased in the form of solution.

ash is rarely found free from impurities. Those most commonly met with are organic matter, sulphate and carbonate of potash, chloride of potassium, silicic acid, alumina, and oxide of lead.*

(*a*) If organic matter is present, the solution of potash is more or less brown, and, on evaporation, leaves a brown residue.

(*b*) Sulphuric acid is detected by diluting the potash with water, supersaturating with nitric or hydrochloric acid, and adding chloride of barium, when, if it is present, the white insoluble sulphate is thrown down.

(*c*) If carbonic acid is present, it causes effervescence when the solution is supersaturated with hydrochloric acid.

(*d*) A little of the diluted solution is supersaturated with nitric acid, and tested with nitrate of silver: a white curdy precipitate, soluble in ammonia, indicates chloride of potassium.

(*e*) Neutralize a small portion with hydrochloric acid, and evaporate to dryness: if the residue is not wholly soluble in hydrochloric acid, silica is probably present.

(*f*) If alumina is present, it will be precipitated when the hydrochloric solution from (*e*) is treated with a *slight* excess of ammonia.

(*g*) If lead be present, the potash will give a dark precipitate or coloration with hydrosulphate of ammonia.

Potash is used chiefly for the purpose of precipitating some of the metallic oxides from their saline solutions, which it does on account of its strong attraction for the acids with which they were in combination. It is employed also for neutralizing acid solutions, decomposing organic compounds, and many other purposes. A solution of potash having a specific gravity of about 1060 (about one part of the solid hydrate dissolved in eight parts of water) is of convenient strength for most analytical purposes. (See Table in Appendix).

658. *Protochloride of Tin* (*Stannous Chloride*), $SnCl_2$.—Protochloride of tin is prepared by boiling metallic tin in strong hydrochloric acid, care being taken that a por-

* When potash is kept in flint-glass bottles, it dissolves oxide of lead from the glass. German or green glass bottles are preferable. Nitrate of potash is sometimes present in the fused potash of commerce.

tion of the metal remains undissolved, as otherwise a little perchloride might be formed: the solution is then diluted with about four times its bulk of water. A few fragments of metallic tin should be kept in the solution, in order to prevent the formation of any perchloride.

Protochloride of tin is employed chiefly as a test for gold and mercury, and also as a dechlorinizing agent, for which purpose it is well adapted, on account of its strong tendency to combine with chlorine.

It occasionally contains traces of lead and iron, which may be detected by adding yellow hydrosulphate of ammonia in excess to the solution and applying heat, when, if pure, the precipitate is wholly redissolved, but if either of those metals is present, a black residue is left, since their sulphides are insoluble in the hydrosulphate.

659. *Solution of Starch*, $C_6H_{10}O_5$.—This is made by mixing 50 grains of white starch with an ounce of water in a mortar, and pouring the mixture by degrees into five ounces of boiling water, with constant stirring. It is employed as a test for iodine, for which purpose small pieces of thread or paper may be steeped in the solution, dried, and kept for use.

660. *Sulphate of Copper* (*Cupric Sulphate*), $CuSO_4$.—This salt is occasionally used as a test for arsenic, and for other purposes: it may be dissolved in ten parts of water. The *ammonio-sulphate of copper*, which is also used in testing for arsenic, is prepared by adding ammonia to the solution of sulphate of copper, until the precipitate at first formed is nearly all redissolved, when the solution is filtered, and kept for use.

661. *Sulphate of Iron* (*Ferrous Sulphate*), $FeSO_4$.—It is advisable to preserve this salt in the solid state, since its solution absorbs oxygen readily from the air; it is employed chiefly in testing for nitric and hydrocyanic acids.

662. *Sulphate of Lime* (*Calcium Sulphate*), $CaSO_4$.—Sulphate of lime being very sparingly soluble in water, is always used in the form of a saturated solution, which is prepared by digesting the sulphate in hot water, stirring it occasionally, and filtering off the clear solution from the undissolved portion. It is used chiefly for distinguishing

baryta from strontia. The solution ought to give an immediate precipitate of sulphate of baryta, when tested with chloride of barium.

663. *Sulphate of Magnesia* (*Magnesium Sulphate*), $MgSO_4$.—This salt is employed as a test for phosphoric acid; a cold saturated solution is convenient for this purpose. It must not give any precipitate with ammonia in presence of chloride of ammonium even on standing.

664. *Sulphate of Manganese* (*Manganous Sulphate*), $MnSO_4$.—A solution of this salt is employed in testing for hypochlorites.

665. *Sulphide of Ammonium;* see *Hydrosulphate of Ammonia.*

666. *Sulphindigotic Acid.*—This substance may be prepared in solution, by dissolving a little indigo in strong sulphuric acid, and diluting the acid solution with water, so as to form a pale blue liquid. It is used chiefly as a test for nitric acid and chlorine, by which it is decomposed, and its colour discharged.

667. *Sulphur* should be tested by gently heating about ten grains in an open porcelain crucible, when no residue should be left after the combustion of the sulphur.

668. *Sulphuric Acid,** H_2SO_4.—Sulphuric acid, as found in commerce, is never pure. The most common impurities are sulphate of lead, nitric acid, or binoxide of nitrogen, and occasionally arsenic, and saline matters.

(*a*) If it contains the first, it will become turbid when diluted with four or five times its bulk of water, owing to the sulphate of lead, which is soluble in the strong acid, being insoluble in the dilute.

(*b*) Nitric acid, or the binoxide of nitrogen, is detected by boiling a small portion tinged with a solution of sulphate of indigo, when, if nitric acid is present, the blue colour will disappear.†

(*c*) Arsenic may be detected by Marsh's test (257), or by mixing the sulphuric acid with an excess of an acid solution of stannous chloride, and heating, when a dark brown precipitate of arsenic is obtained.

* The strength of the acid is indicated by its specific gravity, which should be about 1·84. (See Appendix, Table I.)

† A more delicate test consists in pouring the acid slowly down to the bottom of a test-tube containing solution of sulphate of iron (367).

(*d*) Any fixed saline impurity remains as a residue when a few drops of the acid are evaporated on platinum foil.

669. The uses of sulphuric acid are very numerous. Besides being employed extensively in many branches of manufacture, it is used in the laboratory as a powerful decomposing agent; owing to its strong attraction for bases, nearly all saline compounds are decomposed by it, and its solvent powers are also very great. It is often employed for the purpose of decomposing organic matter; also in the preparation of hydrogen, hydrosulphuric acid, and other gases; as a test for certain metals, and for many other purposes.

670. When *dilute* sulphuric acid is required, it is prepared by mixing together in a beaker, one part of the strong acid with four parts of distilled water, always *adding the acid to the water*, which should be kept constantly stirred, and allowing the precipitated sulphate of lead (if any) to subside, after which the clear liquid may be poured off.

671. *Tartaric Acid*, $H_2C_4H_4O_6$.—Tartaric acid sometimes contains a trace of lime and sulphuric acid, but is usually sufficiently pure for analytical purposes. The lime may be detected by neutralizing a portion with ammonia, and adding oxalate of ammonia; and the sulphuric acid by chloride of barium.

Tartaric acid is used as a test for potash, with which it forms a sparingly soluble bitartrate. Its property of preventing the precipitation of iron and some other metals by the alkalies (448) is occasionally made available in analysis. It should be kept in a solid state, and a solution made when required, as when kept in solution it soon becomes mouldy;* for this purpose the crystallized acid may be dissolved in about three times its weight of water.

672. *Water*.—Pure distilled water is prepared by carefully distilling any of the common kinds of water either in a still or retort, rejecting the first and last portions (67). For many purposes, rain water, when collected at a distance from towns or manufactories, and boiled and filtered, will be found sufficiently pure; but in analytical experiments, distilled water ought always to be used.

* This may be prevented by adding a very minute quantity of carbolic acid, which does not interfere with the uses of tartaric acid in analysis.

673. Before taking it into use, it should be tested with the following reagents :—

(*a*) Litmus and turmeric paper, for free acids and alkalies.

(*b*) Chloride of barium for sulphates.

(*c*) Nitrate of silver for chlorides. The mixture shortly becomes dark-coloured, if organic matter is present.

(*d*) Oxalate of ammonia for lime.

(*e*) Lime water for carbonic acid.

(*f*) Hydrosulphate of ammonia for any metals of the first or second class.

(*g*) When heated on a slip of glass it should leave the very slightest trace of solid residue.

674. *Zinc.*—In order to granulate it, scrap zinc is melted in an iron ladle, and poured from a height of eight or ten feet, in a slender stream, into a pail of water. It is liable to contain a little antimony and arsenic, which may be detected by Marsh's test (257).

The black scales which are left when commercial zinc is dissolved in dilute sulphuric or hydrochloric acid consist of lead; and a little tin may sometimes be detected in the hydrochloric solution by perchloride of mercury (230): these impurities, however, do not interfere with the common use of zinc for evolving hydrogen.

CHAPTER II.

METHODS OF IMPROVISING REAGENTS.

675. For the benefit of analysts who may be situated at a distance from towns where the necessary reagents can be purchased, a few suggestions are here offered as to the readiest methods of supplying the deficiency from sources which are generally available.

The *primary* chemicals, as they may be styled, with which it is indispensable that the analyst should be supplied, are—

Sulphuric Acid (Vitriol).
Carbonate of Soda (Washing Soda).
Chloride of Sodium (Common Salt).
Nitrate of Potash (Saltpetre).

Sulphur (Brimstone).
Ammonia (Hartshorn).
Lime.
Biborate of Soda (Borax).
Ferrocyanide of Potassium (Prussiate of Potash).
Carbonate or Sulphate of Baryta (Heavy Spar, Barytes).
Sulphate of Magnesia (Epsom Salts).
Litmus (Archil).

With the aid of these, and of such common materials as can be everywhere procured, all the reagents absolutely necessary for qualitative analysis can be improvised. These follow in alphabetical order.

Acetate of lead (sugar of lead) may be made by leaving scraps of lead (or shot) in an open bottle, partly covered with vinegar, until the latter is distinctly sweet, when water may be added, and the solution filtered.

Acetic Acid, by distilling common vinegar. Even vinegar itself might, on an emergency, be used instead of acetic acid, especially after boiling with a little wood charcoal, and filtering.

Ammonia.—The most portable form for this reagent is sal-ammoniac, from which it may be prepared by (79). Common smelling-salts (Preston salts, carbonate of ammonia) might be substituted for the sal-ammoniac.

Antimoniate of potash may be prepared by adding powdered antimony (or crude antimony) to nitre melted in a crucible as long as brisk deflagration ensues. The solution is then prepared from this mass as in (622).

It may also be prepared by dissolving antimony or crude antimony in a mixture of four measures of hydrochloric and one measure of nitric acid, adding a large excess of potash, boiling, and filtering.

Baryta.—To prepare the hydrate of baryta, powdered chloride of barium may be added to a strong boiling solution of (caustic) potash or soda, and the solution filtered while hot; on cooling it will deposit crystals of hydrate of baryta, which may be purified by recrystallization. Or the carbonate of baryta (or sulphate of baryta after calcination with charcoal) may be dissolved in dilute nitric acid, the solution evaporated, and the residue of nitrate of baryta calcined in a crucible or iron pot; anhy-

drous baryta is left, which becomes hydrate in contact with water.

Carbonate of Ammonia.—Common smelling salts may be employed. Or if sal-ammoniac and carbonate of soda be distilled with a little water, a solution of carbonate of ammonia would be obtained. Putrefied urine is an excellent source from which to distil carbonate of ammonia.

Chloride of ammonium (sal-ammoniac), by neutralizing hydrochloric acid with ammonia or carbonate of ammonia.

Chloride of barium may be prepared from the carbonate (witherite) or from the sulphate (heavy spar), both of which are used by colour makers and others under the name of barytes. From the former the chloride of barium is made by dissolving in dilute hydrochloric acid and crystallizing. The sulphate of baryta is mixed with one-fourth its weight of charcoal and enough oil to form a paste; strongly calcined in a closed crucible, and the resulting sulphide of barium dissolved in hydrochloric acid, filtered and crystallized.*

Chloride of calcium is prepared by neutralizing dilute hydrochloric acid with chalk.

Cyanide of potassium is made by fusing eight parts of well-dried ferrocyanide of potassium with three parts of dry carbonate of potash in a covered crucible till a drop on the end of an iron rod is white, or nearly so, on cooling.

Hydrochloric Acid (muriatic acid) would be prepared by distilling common salt with sulphuric acid (76).

Hydrosulphate of ammonia may be made with hydrosulphuric acid and ammonia (641).

Hydrosulphuric Acid.—The sulphide of iron required for its preparation is made by throwing a mixture of 10 parts of iron filings with 7 parts of sulphur, by small portions at a time, into a red-hot crucible. The hydrosulphuric acid is then prepared according to (641).

If crude antimony ore (tersulphide of antimony) is procurable, hydrosulphuric acid may be prepared from it by heating with hydrochloric acid in a flask, the gas being washed by passing through water.

* Or the sulphate of baryta may be mixed with two parts of dry carbonate of soda, two parts of charcoal, and twelve parts of dry saltpetre. The mixture is kindled on an iron plate, allowed to burn out, and the fused mass treated with boiling water. Carbonate of baryta is left, which may be washed thoroughly and dissolved in dilute hydrochloric acid.

A mixture of equal weights of sulphur and tallow, gently heated, will also evolve hydrosulphuric acid.

Powdered galena (sulphuret of lead, lead ore) heated with strong hydrochloric acid, will also furnish hydrosulphuric acid.

Lime (quick lime) is easily procurable by calcining egg-shells, oyster-shells, chalk, or marble in a strong fire.

Nitrate of silver (lunar caustic) is obtained by dissolving ordinary silver in nitric acid, evaporating to dryness in a dish, heating moderately for some time till the (blue) nitrate of copper is converted into (black) oxide, dissolving in water, and filtering.

Nitric Acid (aqua fortis), by distilling saltpetre with sulphuric acid (84).

Oxalate of ammonia is prepared by neutralizing oxalic acid with ammonia or carbonate of ammonia. The acid is obtained by dissolving one part of sugar in eight parts of ordinary nitric acid and evaporating gently to crystallization.

Perchloride of iron (muriate of iron) is made by dissolving iron in hydrochloric acid mixed with one-fourth of nitric acid, and expelling the excess of acid by evaporation.

Phosphate of soda is generally procurable at the druggists. A substitute for it is phosphate of ammonia, prepared by mixing three parts of bone-ash (bones burnt white in an open fire) with two parts of strong sulphuric acid and twenty of water, allowing the mixture to stand for a day or two, straining off the sulphate of lime, and mixing the solution with ammonia or carbonate of ammonia till it is alkaline. The solution of phosphate of ammonia is then filtered off, and concentrated by evaporation.

Potash may be obtained by dissolving the carbonate of potash (common potashes, pearlash) in ten parts of water, and adding, by degrees, to the boiling solution, one part of quick-lime, slaked and mixed with water. Allow the mixture to settle in a covered vessel, and draw off the clear solution of potash. The carbonate of potash may be extracted from wood ashes (especially those of young shoots and leaves) by treatment with water; or from calcined argol (cream of tartar); or from the lees of wine, after calcination. Soda is a good substitute for potash,

and may be prepared from carbonate of soda (common washing soda, soda-ash) by the process described above.

Sulphate of iron (copperas, green vitriol) is made by dissolving iron in dilute sulphuric acid and crystallizing.

Sulphate of lime is to be procured almost everywhere as *plaster of Paris.* It may easily be made by adding chalk to diluted sulphuric acid till it is no longer acid.

Tartaric Acid (not absolutely indispensable in analysis) might be made by boiling 4 parts of cream of tartar (argol, bitartrate of potash) with 1 part of chalk, adding to the mixture 2 parts of chalk dissolved in the least possible quantity of hydrochloric acid, filtering through a cloth, washing the precipitate (tartrate of lime) with water, and digesting at a gentle heat with $\frac{3}{5}$ths as much sulphuric acid as there was originally of cream of tartar, diluted with 6 parts of water. The insoluble sulphate of lime is strained off, and the solution crystallized by slow evaporation.

APPENDIX.

ATOMIC WEIGHTS.

Aluminium	Al	27.5	Mercury	Hg	200
Antimony	Sb	122	Molybdenum	Mo	96
Arsenic	As	75	Nickel	Ni	59
Barium	Ba	137	Niobium	Nb	94
Bismuth	Bi	210	Nitrogen	N	14
Boron	B	10.9	Osmium	Os	199
Bromine	Br	80	Oxygen	O	16
Cadmium	Cd	112	Palladium	Pd	106.5
Cæsium	Cs	133	Phosphorus	P	31
Calcium	Ca	40	Platinum	Pt	197.1
Carbon	C	12	Potassium	K	39.1
Cerium	Ce	94.2	Rhodium	Ro	104.3
Chlorine	Cl	35.5	Rubidium	Rb	85.3
Chromium	Cr	52.5	Ruthenium	Ru	104.2
Cobalt	Co	59	Selenium	Se	79.5
Copper	Cu	63.5	Silicon	Si	28
Didymium	Di	96	Silver	Ag	108
Erbium	E	112.6	Sodium	Na	23
Fluorine	F	19	Strontium	Sr	87.5
Gallium	Ga		Sulphur	S	32
Glucinum	Gl	9.2	Tantalum	Ta	182
Gold	Au	196.6	Tellurium	Te	129
Hydrogen	H	1	Thallium	Tl	204
Indium	In	75.6	Thorinum	Th	238
Iodine	I	127	Tin	Sn	118
Iridium	Ir	197.1	Titanium	Ti	50
Iron	Fe	56	Tungsten	W	184
Lanthanium	La	92	Uranium	U	120
Lead	Pb	207	Vanadium	V	51.3
Lithium	L	7	Yttrium	Y	61.7
Magnesium	Mg	24.3	Zinc	Zn	65
Manganese	Mn	55	Zirconium	Zr	89.5

WEIGHTS AND MEASURES.

Apothecaries' Weight.

Pound.		Ounces.		Drachms.		Scruples.		Grains.		French Grammes.
1	=	12	=	96	=	288	=	5760	=	372.96
		1	=	8	=	24	=	480	=	31.08
				1	=	3	=	60	=	3.885
						1	=	20	=	1.295
								1	=	0.0647

Avoirdupois Weight.

Pound.		Ounces.		Drachms.		Grains.		French Grammes.
1	=	16	=	256	=	7000	=	453.25
		1	=	16	=	437.5	=	28.328
				1	=	27.343	=	1.77

Imperial Measure.

Gallon.		Pints.		Fluid Ounces.		Fluid Drachms.		Minims.
1	=	8	=	160	=	1280	=	76,800
		1	=	20	=	160	=	9,600
				1	=	8	=	480
						1	=	60

Weight of Water at 62°, contained in the Imperial Gallon, &c.

			Grains.
1 Imperial Gallon	. . .	= . . .	70,000
1 ,, Pint	. . .	= . . .	8,750
1 ,, Fluid Ounce	. .	= . . .	437.5
1 ,, Fluid Drachm	. .	= . . .	54.7
1 ,, Minim	. . .	= . . .	0.91

Cubic inches contained in the Imperial Gallon, &c.

			Cubic Inches.
1 Imperial Gallon	. . .	= .	277.276
1 ,, Pint	. . .	= .	34.659
1 ,, Fluid Ounce	.	= .	1.7329
1 ,, Fluid Drachm	.	= .	0.2166
1 ,, Minim	. . .	= .	0.0036

FRENCH WEIGHTS AND MEASURES.

Measures of Length.

		English Inches.		Mil.	Fur.	Yds.	Feet.	In.
Millimetre	=	.03937						
Centimetre	=	.39371						
Decimetre	=	3.93710						
Metre	=	39.37100						
Decametre	=	393.71000	=	0	0	10	2	9.7
Hecatometre	=	3937.10000	=	0	0	109	1	1
Kilometre	=	39371.00000	=	0	4	213	4	10.2
Myriometre	=	393710.00000	=	6	1	156	0	6

Measures of Capacity.

		Cubic Inches.		English Imperial Measure. Gall.	Pints.	F.oz.	F.drms.	Min.
Millilitre	=	.06102	=	0	0	0	0	16.3
Centilitre	=	.61028	=	0	0	0	2	43
Decilitre	=	6.10280	=	0	0	3	3	2
Litre	=	61.02800	=	0	1	15	1	43
Decalitre	=	610.28000	=	2	1	12	1	16
Hecatolitre	=	6102.80000	=	22	0	1	4	48
Kilolitre	=	61028.00000	=	220	0	12	6	24
Myriolitre	=	610280.00000	=	2200	7	13	4	48

Measures of Weight.

		English Grains.		*Avoirdupois.* lbs.	Oz.	Drms.
Milligramme	=	.0154				
Centigramme	=	.1543				
Decigramme	=	1.5432				
Gramme	=	15.4323				
Decagramme	=	154.3234	=	0	0	5.65
Hecatogramme	=	1543.2348	=	0	3	8.5
Kilogramme	=	15432.3480	=	2	3	5
Myriogramme	=	154323.4800	=	22	1	2

TABLE OF ANALYTICAL CLASSIFICATION.

Showing the behaviour of Solutions of the Metals with Hydrosulphuric Acid, Ammonium Sulphide, and Ammonium Carbonate, employed successively.—(The rarer metals are printed in italics.)

Elements precipitated from their acid solutions by Hydrosulphuric Acid, as Sulphides.				Bodies precipitated by Ammonium Sulphide.					Bodies not precipitated by Hydrosulphuric Acid, or Ammonium Sulphide. In the presence of Ammonium Chloride on addition of Ammonium Carbonate, are	
Soluble in Ammonium Sulphide, and reprecipitated by Hydrochloric Acid.		Insoluble in Ammonium Sulphide.		As Sulphides.		As Oxides.		As Salts.	precipitated.	not precipitated.
Antimony . . .	Orange.	Mercury . . .	Black or Brownish black.	Nickel . . .	Black.	Aluminium	Soluble in potash.	Baryta, Strontia, Lime	Barium.	Magnesium.
Arsenic . . .	Yellow.	Silver		Cobalt . . .		*Gallium*		in combination with phosphoric, boracic, oxalic, and some other acids.	Strontium.	Potassium.
Tin		Lead		Manganese	Flesh-coloured.	*Glucinum*			Calcium.	Sodium.
Gold	Black.	Bismuth . . .		Iron	Black.	Chromium				*Lithium.*
Platinum . .		Copper		Zinc	White.	*Niobium.*				*Rubidium.*
Iridium . . .		*Cadmium* . .	Yellow.	*Indium* . .	White.	*Thorinum*	Insoluble in potash.	Magnesia.		*Cæsium.*
Molybdenum.	Brown.	*Palladium* . .	Brownish black.	*Uranium* .	Brownish black.	*Yttrium*		in combination with phosphoric acid.		Ammonium.
Tellurium . .	,,	*Rhodium* . . .		*Thallium* .	Black.	*Cerium*				
		Osmium . . .				*Zirconium*				
		Ruthenium .	Brown.			*Titanium*				
						Tantalum				
						Didymium				
						Erbium				
						Lanthanium				
						Thorinum.				

TABLE I.

Showing the Quantity of H_2SO_4 *of sp. gr.* 1.8485, *and of* SO_3, *in* 100 *Parts of dilute Sulphuric Acid, of different Specific Gravities (Ure).*

H_2SO_4.	Sp. Gr.	SO_3.	H_2SO_4.	Sp. Gr.	SO_3.
100	1.8485	81.54	65	1.5390	53.00
99	1.8475	80.72	64	1.5280	52.18
98	1.8460	79.90	63	1.5170	51.37
97	1.8439	79.09	62	1.5066	50.55
96	1.8410	78.28	61	1.4960	49.74
95	1.8376	77.46	60	1.4860	48.92
94	1.8336	76.65	59	1.4760	48.11
93	1.8290	75.83	58	1.4660	47.29
92	1.8233	75.02	57	1.4560	46.48
91	1.8179	74.20	56	1.4460	45.66
90	1.8115	73.39	55	1.4360	44.85
89	1.8043	72.57	54	1.4265	44.03
88	1.7962	71.75	53	1.4170	43.22
87	1.7870	70.94	52	1.4073	42.40
86	1.7774	70.12	51	1.3977	41.58
85	1.7673	69.31	50	1.3884	40.77
84	1.7570	68.49	49	1.3788	39.95
83	1.7465	67.68	48	1.3697	39.15
82	1.7360	66.86	47	1.3612	38.32
81	1.7245	66.05	46	1.3530	37.51
80	1.7120	65.23	45	1.3440	36.69
79	1.6993	64.42	44	1.3345	35.88
78	1.6870	63.60	43	1.3255	35.06
77	1.6750	62.78	42	1.3165	34.25
76	1.6630	61.97	41	1.3080	33.43
75	1.6520	61.15	40	1.2999	32.61
74	1.6415	60.34	39	1.2913	31.80
73	1.6321	59.82	38	1.2826	30.98
72	1.6204	58.71	37	1.2740	30.17
71	1.6090	57.89	36	1.2654	29.35
70	1.5975	57.08	35	1.2572	28.54
69	1.5868	56.26	34	1.2490	27.72
68	1.5760	55.45	33	1.2409	26.91
67	1.5648	54.63	32	1.2334	26.09
66	1.5503	53.82	31	1.2260	25.28

TABLE I.—*Continued.*

H_2SO_4.	Sp. Gr.	SO_3.	H_2SO_4.	Sp. Gr.	SO_3.
30	1.2184	24.46	15	1.1019	12.23
29	1.2108	23.65	14	1.0953	11.41
28	1.2032	22.83	13	1.0887	10.60
27	1.1956	22.01	12	1.0809	9.78
26	1.1876	21.20	11	1.0743	8.97
25	1.1792	20.38	10	1.0682	8.15
24	1.1706	19.57	9	1.0614	7.34
23	1·1626	18.75	8	1.0544	6.52
22	1.1549	17.94	7	1.0477	5.71
21	1.1480	17.12	6	1.0405	4.89
20	1.1410	16.31	5	1.0336	4.08
19	1.1330	15.49	4	1.0268	3.26
18	1.1246	14.68	3	1.0206	2.446
17	1.1165	13.84	2	1.0140	1.63
16	1.1090	13.05	1	1.0074	0.8154

TABLE II.

Showing the Quantity of N_2O_5 *in* 100 *Parts of Liquid Nitric Acid of different Specific Gravities* (*Ure*).

Specific Gravity.	N_2O_5 in 100 parts of the Liquid.	Specific Gravity.	N_2O_5 in 100 parts of the Liquid.	Specific Gravity.	N_2O_5 in 100 parts of the Liquid.
1.5000	79.700	1.4600	68.542	1.4065	57.384
1.4980	78.903	1.4570	67.745	1.4023	56.587
1.4960	78.106	1.4530	66.948	1.3978	55.790
1.4940	77.309	1.4500	66.155	1.3945	54.993
1.4910	76.512	1.4460	65.354	1.3882	54.196
1.4880	75.715	1.4424	64.557	1.3833	53.399
1.4850	74.918	1.4385	63.760	1.3783	52.602
1.4820	74.121	1.4346	62.963	1.3732	51.805
1.4790	73.324	1.4306	62.166	1.3681	51.068
1.4760	72.527	1.4269	61.369	1.3630	50.211
1.4730	71.730	1.4228	60.572	1.3579	49.414
1.4700	70.933	1.4189	59.775	1.3529	48.617
1.4670	70.136	1.4147	58.978	1.3477	47.820
1.4640	69.339	1.4107	58.181	1.3427	47.023

TABLE II.—*Continued.*

Specific Gravity.	N_2O_5 in 100 parts of the Liquid.	Specific Gravity.	N_2O_5 in 100 parts of the Liquid.	Specific Gravity.	N_2O_5 in 100 parts of the Liquid.
1.3376	46.226	1.2212	30.286	1.1051	15.143
1.3323	45.429	1.2148	29.489	1.0993	14.346
1.3270	44.632	1.2084	28.692	1.0935	13.549
1.3216	43.835	1.2019	27.895	1.0878	12.752
1.3163	43.038	1.1958	27.098	1.0821	11.955
1.3110	42.241	1.1895	26.301	1.0764	11.158
1.3056	41.444	1.1833	25.504	1.0708	10.361
1.3001	40.647	1.1770	24.707	1.0651	9.564
1.2947	39.850	1.1709	23.910	1.0595	8.767
1.2887	39.053	1.1648	23.113	1.0540	7.970
1.2826	38.256	1.1587	22.316	1.0485	7.173
1.2765	37.459	1.1526	21.519	1.0430	6.376
1.2705	36.662	1.1465	20.722	1.0375	5.579
1.2644	35.865	1.1403	19.925	1.0320	4.782
1.2583	35.068	1.1345	19.128	1.0267	3.985
1.2523	34.271	1.1286	18.331	1.0212	3.188
1.2462	33.474	1.1227	17.534	1.0159	2.391
1.2402	32.677	1.1168	16.737	1.0106	1.594
1.2341	31.880	1.1109	15.940	1.0053	0.797
1.2277	31.083				

TABLE III.

Showing the Quantity of Hydrochloric Acid in the Liquid Acid of different Specific Gravities (Ure).

Specific Gravity.	HCl.	Specific Gravity.	HCl.	Specific Gravity.	HCl.
1.2000	40.777	1.1822	36.700	1.1620	32.621
1.1982	40.369	1.1802	36.292	1.1599	32.213
1.1964	39.961	1.1782	35.884	1.1578	31.805
1.1946	39.554	1.1762	35.476	1.1557	31.398
1.1928	39.146	1.1741	35.068	1.1536	30.990
1.1910	38.738	1.1721	34.660	1.1515	30.582
1.1893	38.330	1.1701	34.252	1.1494	30.174
1.1875	37.923	1.1681	33.845	1.1473	29.767
1.1857	37.516	1.1661	33.437	1.1452	29.359
1.1846	37.108	1.1641	33.029	1.1431	28.951

Specific Gravity.	HCl.	Specific Gravity.	HCl.	Specific Gravity.	HCl.
1.1410	28.544	1.0919	18.757	1.0457	9.379
1.1389	28.136	1.0899	18.349	1.0437	8.971
1.1369	27.728	1.0879	17.941	1.0417	8.563
1.1349	27.321	1.0859	17.534	1.0397	8.155
1.1328	26.913	1.0838	17.126	1.0377	7.747
1.1308	26.505	1.0818	16.718	1.0357	7.340
1.1287	26.098	1.0798	16.310	1.0337	6.932
1.1267	25.690	1.0778	15.902	1.0318	6.524
1.1247	25.282	1.0758	15.494	1.0298	6.116
1.1226	24.874	1.0738	15.087	1.0279	5.709
1.1206	24.466	1.0718	14.679	1.0259	5.301
1.1185	24.058	1.0697	14.271	1.0239	4.893
1.1164	23.650	1.0677	13.863	1.0220	4.486
1.1143	23.242	1.0657	13.456	1.0200	4.078
1.1123	22.834	1.0637	13.049	1.0180	3.670
1.1102	22.426	1.0617	12.641	1.0160	3.262
1.1082	22.019	1.0597	12.233	1.0140	2.854
1.1061	21.611	1.0577	11.825	1.0120	2.447
1.1041	21.203	1.0557	11.418	1.0110	2.039
1.1020	20.796	1.0537	11.010	1.0080	1.631
1.1000	20.388	1.0517	10.602	1.0060	1.224
1.0980	19.980	1.0497	10.194	1.0040	0.816
1.0960	19.572	1.0477	9.786	1.0020	0.408
1.0939	9.165				

TABLE IV.

Showing the Quantity of Anhydrous Potash (K_2O) *in Solutions of different Specific Gravities* (*Dalton*)

Specific Gravity.	Potash per cent.	Boiling Point.	Specific Gravity.	Potash per cent.	Boiling Point.
1.63	51.2	329°	1.33	26.3	229°
1.60	46.7	290	1.28	23.4	224
1.52	42.9	276	1.23	19.5	220
1.47	39.6	265	1.19	16.2	218
1.44	36.8	255	1.15	13.0	215
1.42	34.4	246	1.11	9.5	214
1.39	32.4	240	1.06	4.7	213
1.36	29.4	234			

TABLE V.

Showing the Quantity of Anhydrous Soda (Na_2O) *in Solutions of different Specific Gravities (Dalton).*

Specific Gravity.	Soda per cent.	Boiling Point.	Specific Gravity.	Soda per cent.	Boiling Point.
2.00	77.8	——°	1.40	29.0	242°
1.85	63.6	600	1.36	26.0	235
1.72	53.8	400	1.32	22.0	228
1.63	46.6	300	1.29	19.0	224
1.56	41.2	280	1.23	16.0	220
1.50	36.8	265	1.18	13.0	217
1.47	34.0	255	1.12	9.8	214
1.44	31.0	248	1.06	4.7	213

TABLE VI.

Showing the Quantity of Ammoniacal Gas (NH_3) *in Aqueous Solutions of different Specific Gravities (Dalton).*

Specific Gravity.	Grains of Ammonia in 100 grains of the liquid.	Boiling Points.	Volumes of gas in one volume of the liquid.
.850	35.3	26°	494
.860	32.6	38	456
.870	29.9	50	419
.880	27.3	62	382
.890	24.7	74	346
.900	22.2	86	311
.910	19.8	98	277
.920	17.4	110	244
.930	15.1	122	211
.940	12.8	134	180
.950	10.5	146	147
.960	8.3	158	116
.970	6.2	173	87
.980	4.4	187	58
.990	2.0	196	28

TABLE VII.

Showing the Quantity of Absolute Alcohol contained in Diluted Alcohol of different Specific Gravities (Fownes).

Sp. Gr. at 60°.	Percentage of real Alcohol.	Sp. Gr. at 60°.	Percentage of real Alcohol.	Sp. Gr. at 60°.	Percentage of real Alcohol.
0.9991	0.5	0.9511	34	0.8769	68
0.9981	1	0.9490	35	0.8745	69
0.9965	2	0.9470	36	0.8721	70
0.9947	3	0.9452	37	0.8696	71
0.9930	4	0.9434	38	0.8672	72
0.9914	5	0.9416	39	0.8649	73
0.9898	6	0.9396	40	0.8625	74
0.9884	7	0.9376	41	0.8603	75
0.9869	8	0.9356	42	0.8581	76
0.9855	9	0.9335	43	0.8557	77
0.9841	10	0.9314	44	0.8533	78
0.9828	11	0.9292	45	0.8508	79
0.9815	12	0.9270	46	0.8483	80
0.9802	13	0.9249	47	0.8459	81
0.9789	14	0.9228	48	0.8434	82
0.9778	15	0.9206	49	0.8408	83
0.9766	16	0.9184	50	0.8382	84
0.9753	17	0.9160	51	0.8357	85
0.9741	18	0.9135	52	0.8331	86
0.9728	19	0.9113	53	0.8305	87
0.9716	20	0.9090	54	0.8279	88
0.9704	21	0.9069	55	0.8254	89
0.9691	22	0.9047	56	0.8228	90
0.9678	23	0.9025	57	0.8199	91
0.9665	24	0.9001	58	0.8172	92
0.9652	25	0.8979	59	0.8145	93
0.9638	26	0.8956	60	0.8118	94
0.9623	27	0.8932	61	0.8089	95
0.9609	28	0.8908	62	0.8061	96
0.9593	29	0.8886	63	0.8031	97
0.9578	30	0.8863	64	0.8001	98
0.9560	31	0.8840	65	0.7969	99
0.9544	32	0.8816	66	0.7938	100
0.9528	33	0.8793	67		

TABLE VIII.

Showing the Specific Gravities of Mixtures of Ether and Alcohol in different proportions (Dalton).

Specific Gravity.	Ether.	Alcohol (sp. gr. 830).	Specific Gravity.	Ether.	Alcohol (sp. gr. 830).
724	100	0	792	40	60
732	90	10	804	30	70
744	80	20	816	20	80
756	70	30	828	10	90
768	60	40	830	0	100
780	50	50			

TABLE IX.

*Showing the Solubility of Salts.**

Bases. / Acids.	Sulphuric.	Phosphoric.	Boracic.	Carbonic.	Silicic.	Arsenious.	Arsenic.	Hydrochloric.	Hydriodic.	Hydrosulphuric.	Nitric.	Chloric.	Oxalic.	Tartaric.	Citric.	Malic.	Succinic.	Benzoic.	Acetic.	Formic.
Potash	1...	1	1	1	1 & 3	1	1...	1...	1	1...	1...	1...	1...	1 & 2	1...	1...	1...	1...	1...	1
Soda	1...	1	1	1	1 ... 3	1	1...	1...	1	1...	1...	1...	1...	1...	1...	1...	1...	1...	1...	1
Ammonia	1...	1	1	1	...	1	1...	1...	1	1...	1...	1...	1...	1 & 2	1...	1...	1...	1...	1...	1
Magnesia	1...	2	2	2	2 & 3	2	2...	1...	1	...	1...	1...	2...	1 ... 2	1...	1 ... 2	1...	1...	1...	1
Lime	1.2.3	2	2	2	2 & 3	2	2...	1...	1	1 ... 2	1...	1...	2...	2...	2...	1 & 2	2...	1 ... 2	1...	1
Baryta	3...	2	2	2	2 & 3	2	2...	1...	1	1...	1...	1...	2...	2...	2...	1...	1 ... 2	1...	1...	1
Strontia	3...	2	2	2	2 & 3	2	2...	1...	1	1...	1...	1...	2...	2...	1...	1...	1...	1...	1...	1
Alumina	1...	2	2	...	2 & 3	2	2...	1...	...	...	1...	1...	2...	1...	1 & 2	2...	...	1...	1...	1 ... 2
Ox. Chromium	1...	2	2	2	2 & 3	...	2...	1...	...	...	1...	1...	1...	1...	1...	...	1...	...	1...	
Ox. Zinc	1...	2	2	2	2 & 3	2	1 & 2	1...	1	2...	1...	1...	2...	2...	2...	1 ... 2	1...	1...	1...	1
Ox. Manganese	1...	2	2	2	2 & 3	...	2...	1...	1	2...	1...	1...	2...	1 ... 2	1...	1...	1...	1...	1...	1
Protox. Iron	1...	2	2	2	2 & 3	2	2...	1...	1	2...	1...	1...	1 ... 2	1 ... 2	1...	1...	1...	...	1...	1
Perox. Iron	1...	2	2	...	2 & 3	...	2...	1...	1	2...	1...	1...	1 ... 2	1...	1...	1...	2...	2...	1...	1
Ox. Nickel	1...	2	2	2	2 & 3	2	2...	1...	...	2...	1...	1...	2...	2...	2...	...	2...	1...	1...	1
Ox. Cobalt	1...	2	2	2	2 & 3	2	2...	1...	...	2...	1...	1...	2...	1...	...	...	2...	1...	1...	1 ... 2
Ox. Antimony	1...2	2	...	...	...	2	2...	1 & 2	...	2...	...	...	1 ... 2	1...	...	...	...	1...	1...	
Subox. Mercury	1...2	2	...	2	...	2	2...	2 ... 3	2	2...	1 & 2	1 & 2	2...	1 ... 2	2...	1...	2...	2...	1 ... 2	1
Perox. Mercury	1...	2	...	2	...	2	2...	1...	2	2...	1...	1...	2...	2...	...	1 & 2	1 ... 2	2...	1...	
Ox. Lead	2...3	2	2	2	3 ...	2	2...	1.2.3	2	2...	1...	1...	2...	2...	2...	2...	1 ... 2	1...	1...	1
Ox. Copper	1...	2	2	2	3 ...	2	2...	1...	2	2...	1...	1...	2...	1...	1...	1...	1...	1 ... 2	1 ... 2	1
Ox. Silver	1...	2	2	2	...	2	2...	3...	3	2...	1...	1...	2...	2...	2...	1...	1...	1...	1...	1
Protox. Tin	1...	2	2	...	...	...	2...	1...	2	2...	...	...	2...	1 ... 2	...	1...	1...	1 ... 2	1...	2
Perox. Tin	1...	2	...	...	...	...	...	1...	...	2...	1...	...	...	...	...	...	2...	...	1...	
Ox. Bismuth	1...	2	2	2	3 ...	...	2...	1 ... 2	2	2...	1 & 2	...	2...	2...	...	...	1...	1...	1...	1

* To ascertain the solubility of any salt, find the name of the base in the upright column, and that of the acid in the line at the top; the number placed at the point where the two rows meet, shows whether the salt formed by their combination is soluble or otherwise. The figure 1 means that it is soluble in water; 2, that it is insoluble in water, but soluble in acids; and 3, that is insoluble in water and acids.

TESTING TABLES,

Showing the Action of Reagents on Oxides and Acids. (Alphabetically arranged.)

1. Metallic Bases. (In Combination.)

Name of Base.	*Hydrosulphuric Acid (Sulphuretted Hydrogen) in an acidified solution.*	*Ammonium Sulphide.*	*Sodium Carbonate.*	*Ammonium Carbonate.*	*Potash.*	*Ammonia.*	*Potassium Ferrocyanide.*	*Blowpipe.*	*Remarks.*
Alumina (see also p. 86).	O	White; insol. in excess.	White; insol. in excess.	White; insol.	White; sol. in excess.	White; insol.	O	Blue with nitrate of cobalt.	These precipitates are insoluble in chloride of ammonium.
Antimony, oxide of (see also p. 76).	Orange red.	Orange red; sol. in excess.	White; sparingly soluble.	White; sp. sol.	White; sp. sol. in excess.	White; insol.	White.	With soda in deoxidizing flame, reduced, and gives off white fumes of oxide.	The chloride decomposed by water.
Baryta (see also p. 96).	O	O	White; insol. in excess.	White; insol.	O	O	O	O	Thrown down immediately by sulphates and sulphate of lime.

Name of Base.	*Hydro-sulphuric Acid, in an acidified solution.*	*Ammonium Sulphide.*	*Sodium Carbonate.*	*Ammonium Carbonate.*	*Potash.*	*Ammonia.*	*Potassium Ferro-cyanide.*	*Blowpipe*	*Remarks.*
BISMUTH, oxide of (see also p. 72.)	Brown-black.	Brown-black; insol. in excess.	White; insol. in excess.	White; insol.	White; insol.	White; insol.	White.	With soda on charcoal, reduced; brittle bead of metal.	Chloride decomposed by water.
CÆSIUM, oxide of.	O	O	O	O	O	O	O	Violet flame (Blue lines in spectrum).	Ppt. with tartaric acid and with $PtCl_4$. Carb. sol. in alcohol.
CADMIUM, oxide of.	Bright yellow.	Bright yellow; insol. in excess.	White; insol. in excess.	White; insol.	White; insol.	White; sol. in excess.	White.	With soda on charcoal reduced and metal volatilized; leaves a reddish-brown deposit.	The yellow sulphide, insol. in hydrosulp. of ammonia, is highly characteristic.
CALCIUM (LIME) (see also p. 98).	O	O	White; insol. in excess.	White; insol.	O	O	O	Radiates a brilliant light. Gives a red colour to the flame.	Oxalate of ammonia causes a white precipitate even in very dilute solutions.
CERIUM, oxide of.	O	White.	White.	White; sp. sol.	White; insol. yellow in air.	White; insol.	O	Borax-bead, outer; yellow-red: inner; colourless.	Ppt. by oxalic, also by saturated K_2SO_4.

Chromium, oxide of (see also p. 87).	O	Green.	Green.	Green.	Green; sol. in excess.	Green; insol.	O	Emerald green with fluxes.	The oxide when fused with nitre, gives chromate of potash.
Cobalt, oxide of (see also p. 95).	O	Black.	Pink; insol. Bluish on boiling.	Pink; sp. sol. in ex. solution purple.	Blue, becoming greenish; dirty red on boiling.	Blue; sol. in ex. forming a brownish-red sol.	Pale green or grey.	Blue glass, with borax in both flames.	Readily distinguished with the blowpipe.
Copper, oxide of (see also p. 73).	Black.	Black.	Greenish blue, becoming dark brown on boiling.	Greenish blue, sol. in ex., forming deep blue solution.	Pale blue, becoming dark brown when boiled.	Pale blue sol. in ex. forming rich blue solution.	Mahogany coloured; insol.	With soda on charcoal, reduced. With borax and mic. salt in outer flame, green; in inner flame red.	Precipitated in the metallic state by clean iron.
Didymium, oxide of.	O	White.	White; insol.	White; insol.	White; insol.	White; insol.	O	Micr. salt inner, violet outer O	Pink ppt. with saturated K_2SO_4; characteristic absorption spectrum.
Gallium, oxide of.	O	White.	White.	White.	White; sol.	White; sp. sol.	White; in acid sols.	Bright violet band in spectrum.	At present imperfectly studied.
Glucina.	O	White; insol. in excess.	White; sp. sol.	White; sol. in ex. Repptd. by boiling.	White; sol. in ex.	White; insol.	O	With nitrate of cobalt, dark grey or black.	Glucina dissolves in cold solution of carb. ammonia, and is thrown down on boiling.

Name of Base.	*Hydro-sulphuric Acid in an acidified solution.*	*Ammonium Sulphide.*	*Sodium Carbonate.*	*Ammonium Carbonate.*	*Potash.*	*Ammonia.*	*Potassium Ferrocyanide.*	*Blowpipe.*	*Remarks.*
GOLD, teroxide of.	Black.	Brown black; sol. in excess.	O	Yellow; insol.	Yellowish brown.	Yellow; insol.	O	Reduced.	Thrown down in the form of a brown metallic powder by proto-sulph. of iron.
INDIUM, oxide of.	O	White; sol. on heating.	White.	White; sol. in ex. Repptd. by boiling.	White; insol.	White; insol.	White.	Violet flame. Blue lines in spectrum.	H_2S in acetic solutions a fine yellow ppt.
IRIDIUM, sesqui-oxide of.	Slight brown.	Brown; sol.	Brown red; sp. sol.	Bleaches the solution.	Slight brown. The solution becomes first colourless, and subsequently bluish.	As with potash. On exposure to the air, a slight blue precipitate falls.	Solution slowly discoloured.	Reduced.	The solutions have a deep brown colour.
IRON, protoxide of (see also p. 91).	O	Black.	White, then green, and ultimately rust-coloured.	As with carbonate of soda.	White; becoming green, and on standing, rust-coloured.	As with potash; but becoming brown more rapidly.	White; instantly changing to light blue.	With the fluxes in the outer flame, brownish-yellow; in the inner flame, light green.	Deep blue precipitate with ferridcyanide of potassium.

Iron, peroxide of (see also p. 92).	Yellowish-white precipitate of sulphur.	Black.	Rust-coloured.	Rust-coloured.	Rust-coloured.	Rust-coloured.	Deep blue.	As the protoxide.	Black with infusion of gall-nuts.
Lanthanium, oxide of.	O	White.	White.	White; insol.	White; insol.	White.	O	O	Acetate + NH_3 in ex. washed and + powd. iodine. Blue colour.
Lead, oxide of (see also p. 68).	Black.	Black.	White; insol.	White; insol.	White; sol.	White; insol. None at first with the acetate.	White.	With soda on charcoal, reduced; yellow deposit also formed on the charcoal.	Precipitd. by soluble sulphates, and the precip. blackened by hydrosulphate of ammonia. Bright yellow with chromate of potash and iodide of potassium.
Lithia.	O	O	Faint white in concentrated sol.	As carb. soda.	O	O	O	Gives red colour to the flame.	Phos. of soda and evapn. gives a white precipitate.
Magnesia (see also p. 99).	O	O	White; insol. on boiling.	White; insol. on boiling.	White; insol.	White; insol.	O	Light pink with nitrate of cobalt.	Crystalline precipit. with phosphate of soda, and ammonia. The carbonate and hydrate sol. in chloride of ammonium.

Name of Base.	*Hydro-sulphuric Acid in an acidified solution.*	*Ammonium Sulphide.*	*Sodium Carbonate.*	*Ammonium Carbonate.*	*Potash.*	*Ammonia.*	*Potassium Ferrocyanide.*	*Blowpipe.*	*Remarks.*
Manganese, protoxide of (see also p. 90).	O	Flesh-coloured.	White; insol.	White; insol.	White; becoming brown.	White; becoming brown.	White.	With soda, a green bead. With borax in outer flame an amethyst bead, which loses its colour in the reducing flame.	The presence of ammoniacal salts prevents more or less completely the precipitation of manganese by the alkalies.
Mercury, suboxide of (see also p. 70).	Black.	Black.	Dark grey.	Dark grey.	Black; insol.	Black; insol.	White.	Mixed with soda, and heated in a tube; the metal sublimes.	White precipitate with chlorides blackened by ammonia. Volatd. or decompd. by heat.
Mercury, peroxide of (see also p. 71).	White, turning to black.	White, turning to black.	Reddish-brown; insol.	White; insol.	Yellow; insol.	White; insol.	White.	As the protoxide.	Volatilized or decomposed by heat. Beautiful scarlet with iodide of potassium.
Molybdenum, oxides of.	Brown-black, slowly formed.	Yellowish-brown; sol.	Brown; insol.	Brown; sol.	Brown-black; insol.	Brown-black; insol.	Brown, with the binoxide.	With microcosmic salt in outer flame, a green glass.	Most readily distinguished by the blowpipe.

Nickel, oxide of (see also p. 93).	O	Black.	Pale green; insol.	Pale green; soluble, forming a blue solution.	Pale green; insol.	Pale green; soluble, forming a blue solution.	Pale green.	With soda on charcoal reduced to a magnetic powder. With borax and mic. salt in outer flame, red glass becoming grey in inner flame.	Potash throws down a pale green precipitate from the ammoniacal solution.
Osmium, binoxide of.	Yellowish-brown, slowly formed.	Yellowish-brown; insol.	Black, slowly formed. Bluish solution.	Brown after some time.	Black on boiling.	Brown after some time.	O	Osmium is characterized by forming, when heated in the air, osmic acid, which is volatile, and has a very disagreeable smell, causing much inconvenience to the eyes and nose.	
Palladium, protoxide of.	Black.	Black; insol.	Brown; sol. Reprecipitated on boiling.	Solution decolorized, but no precipitate	Yellowish-brown; sol.	Yellowish-brown;	O	Reduced.	Yellowish white with solution of cyanide of mercury.
Platinum, oxide of.	Brownish-black, formed slowly.	Brownish-black; sol. in large excess.	Yellow with carbonate of potash.	Yellow.	Yellow.	Yellow.	O	Reduced.	Yellow with chloride of ammonium, which is converted by heat into spongy platinum.
Potash (see also p. 100).	O	O	O	O	O	O	O	Violet flame.	White crystalline precipitate with tartaric acid. Yellow with bichloride of platinum.

Name of Base.	*Hydro-sulphuric Acid in an acidified solution.*	*Ammonium Sulphide.*	*Sodium Carbonate.*	*Ammonium Carbonate.*	*Potash.*	*Ammonia.*	*Potassium Ferro-cyanide.*	*Blowpipe.*	*Remarks.*
RHODIUM, sesquioxide of.	Brown, formed slowly.	Brown; insol.	Yellowish after a time.	Yellowish after a time.	Yellowish-brown on boiling.	Yellowish after a time.	Dark orange.	Reduced.	Many of the compounds have a rose-colour.
RUBIDIA.	O	O	O	O	O	O	O	Violet flame. Red lines in spectrum.	Precipitate with tartaric acid and $PtCl_4$. Carb. insol. in alcohol.
RUTHENIUM, sesquioxide of.	Brown; blue solution.	Brown-black; sp. sol.	Black.	Black.	Black.	O	O		KScy., red, purple, and violet on heating.
SILVER, oxide of (see also p. 67).	Black.	Black.	White; insol.	White. sol.	Pale brown; insol.	Pale brown; sol.	White.	Reduced.	White curdy precipitate with hydrochloric acid and chlorides, which is sol. in ammonia, and insol. in nitric acid.
SODA (see also p. 101).	O	O	O	O	O	O	O	Yellow flame.	The only salt which precipitates soda, is the antimoniate of potash. Evaporated with bichloride of platinum, gives yellow needles.

Strontia (see also p. 97).	O	O	White; insol.	White; insol.	O	O	O	Carmine flame.	White precipitate with sulphates. Burnt with alcohol, gives carmine flame.
Thallium, oxide of.	O	Black.	White in conc. sol.	White in conc. sol.	O	O	O	Green flame. Green band in spectrum.	White precipitate with HCl, yellow with KI.
Thorinum, oxide of.	O	White.	White.	White: sol. in ex. pptd. by heating.	White; insol.	White.	O	O	Precipitated by saturated K_2SO_4, by HF, and by oxalic.
Tin, protoxide of (see also p. 74).	Brown-black.	Brown-black; sol. in yellow hydros.	White; insol.	White; insol.	White; sol.	White; insol.	White.	With soda in reducing flame a malleable bead of metallic tin.	Zinc throws down the metal as a grey sponge.
Tin, peroxide of (see also p. 75).	Yellow.	Yellow; sol.	White; insol.	White; insol.	White; sol.	White; sp. sol.	White.	Reduced with soda.	The behaviour with hydrosulphate of ammonia and the blowpipe is characteristic.
Uranium, sesquioxide of.	O (sulphur).	Black.	Yellow; sol.	Yellow; sol.	Yellow; insol.	Yellow; insol.	Red-dish-brown.	Yellow glass with borax.	When the precipitate with ammonia is heated, it is converted into the green protoxide.

Name of Base.	*Hydrosulphuric Acid in an acidified solution.*	*Ammonium Sulphide.*	*Sodium Carbonate.*	*Ammonium Carbonate.*	*Potash.*	*Ammonia.*	*Potassium Ferrocyanide.*	*Blowpipe.*	*Remarks.*
VANADIUM, binoxide of.	O	Brown-black; sol. in excess, forming a purple solution.	Dirty white.	Grey, passing to brown.	Greyish white.	Brown.	Yellow.	With borax, yellow in outer flame; in the inner, brown, becoming green when cold.	Many of the solutions have a blue colour.
YTTRIA.	O	White.	White; sp. sol.	White; sp. sol.	White; insol.	White; insol.	White.	Nothing characteristic.	Copious white with oxalic acid.
ZINC, oxide of (see also p. 89).	O	White.	White; insol.	White; sol.	White; sol.	White; sol.	White.	With soda on charcoal gives a white sublimate of oxide, which is yellow when hot. With nit. cobalt, green.	Behaviour with hydrosulphate of ammonia characteristic.
ZIRCONIA.	O	White.	White after a time.	White; sol.	White; insol.	White; insol.	White.	Bright light.	Oxalic acid gives a white precipitate.

2. Metallic Oxides having acid properties.

Acids (in combination).	*Hydrosulphuric acid, in acidified solutions.*	*Ammonium Sulphide.*	*Barium Chloride (in alkaline salts of the acids).*	*Silver Nitrate (in alkaline salts of the acids).*	*Calcium Nitrate (in alkaline salts of the acids).*	*Hydrochloric acid.*	*Remarks.*
Antimonic acid.	Orange.	Orange; sol.	White.	White.	White.	White; sol. in excess.	Insoluble in water and nitric acid. Soluble in hydrochloric acid, from which it is precipitated on the addition of water.
Arsenious acid (see also p. 77).	Yellow; soluble in alkalies and alkaline sulphides.	Yellow; sol.	White.	Pale yellow.	White.	O	Volatilizes at a low heat, and condenses in octahedral crystals The best tests are Marsh's and Reinsch's. (See p. 81).
Arsenic acid (see also p. 86).	Yellow; soluble in alkalies and alkaline sulphides.	Yellow; sol.	White.	Reddish-brown.	White.	O	Heated with black flux, gives metallic arsenic.
Chromic acid.	Reduced to oxide, with precipitation of sulphur.	Green.	Yellow.	Red.	Yellow in concentrated solutions.	Reduced to oxide, with evolution of chlorine, on boiling.	Is decomposed by heat and by deoxidizing agents, into oxide of chromium. Salts of lead throw down a yellow precipitate.

Acids (in combination).	*Hydrosulphuric acid, in acidified solutions.*	*Ammonium Sulphide.*	*Barium Chloride (in alkaline salts of the acids).*	*Silver Nitrate (in alkaline salts of the acids).*	*Calcium Nitrate (in alkaline salts of the acids).*	*Hydrochloric acid.*	*Remarks.*
MANGANIC ACID.	The same.	Flesh-coloured.	Green.	Black (oxide).	Black.	Solution becomes red, and chlorine is evolved.	Converted by acids into permanganic acid and peroxide of manganese; the colour of the solution changing from green to red.
MOLYBDIC ACID.	Brown.	Brown; sol.	White.	White.	White.	White.	With microcosmic salt before the blowpipe, gives a dark blue glass, which becomes green on cooling. When strongly heated, molybdic acid volatilizes and condenses in crystals.
PERMANGANIC ACID.	Red becomes colourless; deposits S.	Buff ppt.	O	Red ppt.	O	Red becomes colourless on boiling. Cl evolved.	Red solutions changed to green by alkalies.
TUNGSTIC ACID.	Slight turbidity.	Brown; sol.	White.	White.	White.	White; insoluble.	Does not volatilize when heated. Has a pale yellow colour, and is insoluble in water and acids. Blue with hydrochloric acid and zinc.
VANADIC ACID.	Grey.	Brown; sol.	Orange.	Yellow.	O	Chlorine evolved.	When treated with hydrochloric acid, the mixture is capable of dissolving gold-leaf. Vanadic acid in solution is readily deoxidised, forming a blue liquid.

3. Non-metallic Acids.

Acids (neutralized).	*Barium Nitrate.*	*Silver Nitrate.*	*Calcium Nitrate.*	*Lead Acetate.*	*Remarks.*
Boracic acid (see also p. 113).	White.	White.	White.	White.	Slightly volatile in the presence of aqueous vapour. Turns turmeric-paper brown, and blue litmus port-wine colour. Gives green colour to the flame of alcohol.
Bromic acid.	White.	White.	O	White.	The bromates are decomposed by heat into bromides and oxygen. Sulphuric acid disengages bromine.
Carbonic acid (see also p. 115).	White.	White.	White.	White.	The carbonates are readily decomposed by acids, carbonic acid gas being given off with effervescence, which, when passed into lime-water, gives a white precipitate.
Chloric acid (see also p. 109).	O	O	O	O	Detonate when heated with strong sulphuric acid. At a red heat chlorates are converted into chlorides, oxygen being given off.
Chlorous acid.	O	Yellowish precipitate. Sol. in much water.	O	Yellow precipitate.	Transient red colour with $FeSO_4$ and H_2SO_4 in dilute solutions.
Hydriodic acid (see also p. 111).	O	Pale Yellow.	O	Bright yellow.	The iodides evolve iodine when heated with nitric or sulphuric acid. With chlorine water and starch, they give a dark purple precipitate.
Hydrobromic acid.	O	Yellowish.	O	White.	The bromides, when heated with nitric or sulphuric acid, evolve bromine.

Acids (neutralized).	*Barium Nitrate.*	*Silver Nitrate.*	*Calcium Nitrate.*	*Lead Acetate.*	*Remarks.*
HYDROCHLORIC ACID (see also p. 109).	O	White.	O	White.	The chlorides, when heated with sulphuric acid and peroxide of manganese, evolve chlorine.
HYDROCYANIC ACID.	O	White.	O	White.	With a mixture of a protosalt and persalt of iron, the alkaline cyanides give a precipitate of Prussian blue.
HYDROFERRICYANIC ACID.	O	Brown.	O	O	Blue precipitate with $FeSO_4$. Brown colour with Fe_2Cl_6.
HYDROFERROCYANIC ACID.	White.	White.	White.	White.	Blue precipitate with Fe_2Cl_6.
HYDROFLUORIC ACID.	White.	O	White.	O	The fluorides, when moistened with sulphuric acid, give off fumes which corrode glass.
HYDROFLUOSILICIC ACID.	White cryst.	O	O	O	Cryst. ppt. with KNO_3. Corrodes glass when heated with H_2SO_4.
HYDROSELENIC ACID.	O	Black.	O	Black.	The selenides, when heated in the outer flame of the blowpipe, evolve the odour of selenium, resembling that of putrid horse-radish.
HYDROSULPHURIC ACID (see also p. 106).	O	Black.	O	Black.	Most of the sulphides, when treated with an acid, evolve hydrosulphuric acid, which smells like rotten eggs.
HYPOCHLOROUS ACID (see also p. 110).	O	White.	O	White ppt. changing to brown.	Black ppt. with $MnSO_4$. Cl evolved with H_2SO_4.

Hypophosphorous acid.	O	White, changing to black.	O	O	White ppt. with $HgCl_2$. Odour of PH_3 with Zn and H_2SO_4.
Hyposulphurous acid.	White.	White, becoming brown.	O	White.	The hyposulphites are decomposed by hydrochloric acid; sulphur is precipitated, and sulphurous acid set free.
Hyposulphuric acid.	O	O	O	O	The hyposulphates are decomposed without deposition of sulphur, when boiled with hydrochloric acid; sulphurous and sulphuric acid are formed.
Iodic acid.	White.	White.	White.	White.	The iodates are decomposed by heat into iodides and oxygen.
Metaphosphoric acid.	White; slowly sol. on boiling.	White.	O unless very concentd.	White, fusible on boiling.	Metaphosphates become orthophosphates when fused with Na_2CO_3.
Nitric acid (see also p. 103).	O	O	O	O	When mixed with sulphuric and hydrochloric acids, the nitrates dissolve gold-leaf. With copper filings and sulphuric acid, orange fumes are given off.
Nitrous acid.	O	White; sol. on boiling.	O	O	Red fumes with dil. H_2SO_4. Blue with KI, starch and dil. H_2SO_4. Brown with $FeSO_4$.
Perchloric acid.	O	O	O	O	The perchlorates are resolved by heat into chlorides and oxygen. They are not decomposed in the cold by hydrochloric or sulphuric acid; thus differing from the chlorates.
Periodic acid.	White.	Yellow.	White.	White.	Periodates, heated to redness, become iodides.
Phosphoric acid (Tribasic) (see also p. 107)	White.	Yellow.	White.	White.	The soluble phosphates give with salts of magnesia, when ammonia is present, a white crystalline precipitate.

Acids (neutralized).	*Barium Nitrate.*	*Silver Nitrate.*	*Calcium Nitrate.*	*Lead Acetate.*	*Remarks.*
PHOSPHOROUS ACID.	White.	White, becoming brown.	White.	White.	The hydrated phosphites are decomposed when heated in a tube; hydrogen or phosphuretted hydrogen is given off, and phosphates are formed.
PYROPHOSPHORIC ACID.	White.	White.	White.	White.	$MgSO_4$ gives a ppt. sol. in excess, repptd. by boiling. Pyrophosphates become orthophosphates when fused with Na_2CO_3.
SELENIC ACID.	White.	White.	White.	White.	The seleniates are decomposed by boiling with hydrochloric acid; chlorine is evolved, together with selenious acid.
SELENIOUS ACID.	White.	White.	White.	White.	Metallic zinc or sulphurous acid causes the precipitation of selenium from acidified solutions of the selenites.
SILICIC ACID (see also p. 114).	White.	Pale yellow.	White.	White.	When a soluble silicate is evaporated to dryness with hydrochloric acid, it is decomposed, and the silica remains insoluble.
SULPHURIC ACID (see also p. 104).	White.	White crystalline.	White crystalline.	White.	Most of the sulphates, when heated with charcoal, are converted into sulphides, which, when moistened with hydrochloric acid, evolve hydrosulphuric acid.
SULPHUROUS ACID.	White.	White.	White.	White.	The sulphites are decomposed by hydrochloric acid, sulphurous acid being given off, without the deposition of sulphur.

4. Organic Acids.

Acids (neutralized).	Calcium Chloride.	Ferric Chloride.	Barium Nitrate.	Silver Nitrate.	Lead Acetate.	Remarks.
ACETIC ACID. (see also p. 117).	O	Red colour.	O	White crystalline in concentrated solutions.	O	The acetates when warmed with sulphuric acid, give off the smell of vinegar.
BENZOIC ACID (see also p. 122).	O	Brownish-yellow.	O	Crystalline in concentrated neutral solutions.	White in concentrated neutral solutions.	Solutions of the benzoates, when treated with sulphuric acid, give a crystalline precipitate of benzoic acid.
CITRIC ACID (see also p. 121).	White.	O	White.	White.	White.	With protonitrate of mercury, a white precipitate, which becomes grey.
FORMIC ACID.	O	Red colour.	O	White, becoming black, especially when warmed.	O	The formiates when warmed with sulphuric acid, do not blacken, and give off carbonic oxide gas.
MALIC ACID.	White on the addition of alcohol.	O	White.	White; becoming grey.	White precipitate, melts in boiling water.	Malate of lead dissolves in hot dilute acetic acid, and crystallizes on cooling in fine needles.
OXALIC ACID (see also p. 116).	White.	O	White crystalline.	White.	White.	Neither the acid nor the oxalates are blackened by strong sulphuric acid, but give off carbonic acid and carbonic oxide gases.

Acids (neutralized).	*Calcium Chloride.*	*Ferric Chloride.*	*Barium Nitrate.*	*Silver Nitrate.*	*Lead Acetate.*	*Remarks.*
SUCCINIC ACID.	O	Reddish-brown.	O	White on standing.	White.	A mixture of chloride of barium, ammonia, and alcohol, gives a white precipitate of succinate of baryta.
TARTARIC ACID (see also p. 119).	White.	O	White.	White.	White.	Added in excess to potash, gives a crystalline precipitate of the bitartrate.

LIST OF SALTS, &c.

WHICH MAY BE EXAMINED FOR PRACTICE.

I. *Exercises in the use of the Blowpipe* (*p.* 52).

(*a.*) *One constituent to be detected.*

1. Sulphur.
2. Arsenious acid.
3. Calomel.
4. Chloride of ammonium.
5. Oxide of zinc.
6. Binoxide of tin.
7. Oxide of copper.
8. Oxide of lead.
9. Peroxide of iron.
10. Oxide of nickel.
11. Oxide of cobalt.
12. Oxide of chromium.
13. Binoxide of manganese.
14. Carbonate of soda.
15. Bicarbonate of potash.
16. Carbonate of lime.
17. Carbonate of baryta.
18. Silica.
19. Boracic acid.
20. Charcoal.

(*b.*) *Two constituents to be detected.*

21. Sulphate of iron.
22. Trisnitrate of bismuth.
23. Sulphide of arsenic.
24. Vermilion.
25. Iodide of lead.
26. Chlorate of potash.
27. Chloride of sodium.
28. Fluoride of calcium.
29. Sulphide of copper.
30. Sulphate of zinc.
31. Pipe clay.
32. Sulphate of magnesia.
33. Argentiferous lead.*
34. Sulphide of antimony.
35. Iron pyrites.
36. Chromate of potash.
37. Nitrate of strontia.
38. Sulphate of baryta.
39. Borax.
40. Alloy of lead and antimony.†
41. Arsenite of copper.
42. Acetate of lead.

(*c.*) *Three constituents to be detected.*

43. Potash-alum.
44. Ammonia-alum.
45. Tartar-emetic.
46. Arsenical pyrites.

* A pound of lead should be fused with a little borax, and 200 grs. of silver thoroughly dissolved in it. The alloy is then granulated by pouring into water, or better, cast into a thin plate, on a stone, and cut up into fragments.

† An alloy of 4 lead and 1 antimony may be used.

II. *Exercises in Qualitative Analysis.*

(*a.*) *Simple Salts, &c., soluble in Water* (*p.* 127).

1. Chloride of barium.
2. Sulphate of soda.
3. Chloride of ammonium.
4. Sulphate of magnesia.
5. Chloride of calcium.
6. Nitrate of strontia.
7. Sulphate of chromium.
8. Sulphate of zinc.
9. Sulphate of manganese.
10. Protosulphate of iron.
11. Perchloride of iron.
12. Sulphate of nickel.
13. Nitrate of cobalt.
14. Protonitrate of mercury.
15. Perchloride of mercury.
16. Acetate of lead.
17. Sulphate of copper.
18. Nitrate of silver.
19. Phosphate of soda.
20. Iodide of potassium.
21. Biborate of soda.
22. Nitrate of potash.
23. Chlorate of potash.
24. Carbonate of soda.
25. Arsenious acid.

(*b.*) *Simple Salts, &c., insoluble in Water, but soluble in Acids* (*p.* 127).

26. Carbonate of magnesia.
27. Phosphate of lime.
28. Carbonate of baryta.
29. Metallic zinc.
30. Carbonate of strontia.
31. Sulphide of iron.
32. Protoxide of lead.
33. Sulphide of antimony.
34. Black oxide of copper.
35. Carbonate of lime.
36. Oxide of bismuth.
37. Metallic tin.

(*c.*) *Simple Salts, &c., insoluble in Water and Acids* (*p.* 131).

38. Sulphate of baryta.
39. Sulphate of strontia.
40. Chloride of lead.
41. Sulphate of lead.
42. Silica.
43. Chloride of silver.
44. Fluoride of calcium.
45. Silicate of alumina (pipe clay).

(*d.*) *Mixed Salts, &c., soluble in Water* (*p.* 147).*

46. { Sulphate of copper. / Sulphate of iron. }
47. { Nitrate of lead. / Nitrate of silver. }
48. { Sulphate of manganese. / Sulphate of magnesia. }
49. Alum.
50. Phosphate of soda and ammonia.
51. Tartar-emetic.
52. { Carbonate of soda. / Sulphite of soda. }
53. Commercial cyanide of potassium (containing carbonate of potash).
54. { Biborate of soda. / Chloride of ammonium. }

* The constituent placed first should always form the greater part of the mixture.

55. Chlorate of potash. Chloride of sodium.
56. Iodide of potassium. Carbonate of potash.
57. Sulphate of copper. Sulphate of iron. Sulphate of nickel.
58. Sulphate of zinc. Sulphate of copper.
59. Chrome alum.
60. Chloride of barium. Nitrate of strontia.

(*e.*) *Mixed Salts, &c., not entirely soluble in Water, but Soluble in Acids* (*p.* 147).

61. Carbonate of lime. Carbonate of magnesia.
62. Binoxide of manganese. Peroxide of iron. Carbonate of baryta.
63. Sulphide of iron. Arsenious acid.
64. Peroxide of iron. Oxide of cobalt. Oxide of nickel.
65. Calomel. Corrosive sublimate.
66. Acetate of lead. Verdigris.
67. Sulphide of zinc. Sulphide of lead. Sulphide of iron.
68. Peroxide of iron. Binoxide of manganese. Phosphate of lime. Arsenious acid
69. Sulphide of antimony. Sulphide of arsenic. Sulphide of lead.
70. Oxalate of ammonia. Sulphate of lime.
71. Chloride of lime (bleaching powder).
72. Brass.
73. German silver.
74. Phosphate of lime. Alum.

(*f.*) *Mixed Salts, &c., not entirely soluble in Water or Acids* (*p.* 147.)

75. Sulphate of baryta. Sulphate of lime.
76. Pipe clay. Chrome iron ore.
77. Gunpowder.
78. Sulphide of lead. Binoxide of tin. Silica. Fluor spar.
79. Sulphate of lead. Chloride of silver.
80. Vermilion. Red lead.
81. Prussian blue. Carbonate of baryta.
82. Bronze.
83. Pewter.
84. Type metal.
85. Fusible metal.
86. Crown glass.
87. Flint glass.
88. Red earthenware.

GLOSSARY OF CHEMICAL TERMS.*

Absorption, from *absorbeo*, to suck up; the act of imbibing a liquid.

Acetic Acid, from *acetum*, vinegar; the acid contained in vinegar.

Aeriform, from *ἀήρ*, the air, and *forma*, a form; having the form or properties of air.

Affinity, from *ad*, to, and *finis*, a boundary; relationship; the force which causes particles of dissimilar kinds of matter to combine together, so as to form new matter.

Albumen, -inous, from *albumen*, the white of an egg; an important animal principle. The white of an egg consists chiefly of albumen and water, contained in a cellular tissue.

Alcohol, from an Arabic word; the intoxicating principle of spirituous liquors.

Alkali, a soluble body, with a hot caustic taste, which possesses the power of destroying or neutralizing acidity. The term is derived from the Arabic article, *al*, and *kali*, the Arabic name of a plant, from the ashes of which one of the most important alkalies (potash) is obtained.

Amalgam, from *ἅμα*, together, and *γαμέω*, to marry, a term signifying the union of any metal with mercury, which has the property of dissolving several of the metals.

Amorphous, from *ά*, not, and *μορφή*, a form; not possessing any regular form.

Analogue, that which is the counterpart of another.

Analogy, -ical, and -ous, from *ἀνὰ*, among, and *λόγος*, a relation or proportion; a likeness or resemblance between things, with regard to their circumstances or effects.

Analysis, from *ἀνὰ*, among, and *λύω*, to loosen; the separation of a substance into its component parts.

Angle, from *angulus*, a corner; the inclination of two straight lines to each other, which meet together, but are not in the same straight line.

* Many of the definitions are taken almost verbatim from Daniell's Chemical Philosophy.

Anhydrous, from ἀ, not, and ὕδωρ, water; containing no water.

Antiseptic, from ἀντὶ, against, and σήπω, to putrefy; possessing the power of preventing or retarding putrefaction.

Aqua Regia, *i.e.*, Regal Water, a mixture of nitric and hydrochloric acids; so called from its property of dissolving gold, which was held by the alchemists to be the king of the metals.

Aqueo, from *aqua*, water; when prefixed to a word denotes that water enters into the composition of the substance which it signifies, as *aqueo-sulphuric acid* [*Obsolete.*]

Atmolysis, from ἀτμὸς, vapour, and λύω, to loosen; the separation of gases by diffusion through porous substances.

Atmosphere, from ἀτμὸς, vapour, and σφαῖρα, a sphere; commonly used to denote the sphere of air which surrounds the globe.

Atom, -ic, from ἀ, not, and τέμνω, to cut; a minute particle of matter, not susceptible of further division.

Attraction, -ive, from *ad*, to, and *traho*, to draw; the tendency which bodies have to approach or unite with each other.

Azote, from ἀ, not, and ζωὴ, life; another name for nitrogen; so called because it is incapable of supporting respiration.

Barium, from βαρὺς, heavy; the metallic base of baryta.

Barometer, from βάρος, weight, and μέτρον, a measure; an instrument for measuring the varying pressure of the atmosphere.

Baryta, a compound of oxygen and the metal *barium*, possessing alkaline properties.

Basylous. Capable of being substituted for the hydrogen in acids, to form salts.

Bibulous, from *bibo*, to drink; that which has the property of drinking in, or absorbing moisture.

Boron, a dark, olive-coloured elementary substance, obtained from boracic acid, insoluble in water, and a non-conductor of electricity.

Bromine, from βρῶμος, a strong odour; an elementary liquid of a reddish-brown colour and suffocating smell; in chemical properties it strongly resembles chlorine.

Caloric, from *calor*, heat; an imaginary fluid substance, supposed to be diffused through all kinds of matter, and the sensible effect of which is called *heat.*

Capillary, from *capillus*, a hair; resembling, or having the tubular form of hairs.

Capsule, from *capsula*, a little chest; a small shallow cup.

Carbon, from *carbo*, a coal; the chemical name for charcoal.

Caustic, from *καίω*, to burn; possessing the power of burning.

Chemistry, -ical, from an Arabic word, signifying the knowledge of the substance or constitution of bodies; the science whose object it is to examine the constitution of bodies.

Chlorine, from *χλωρὸς*, green; a greenish coloured elementary gas, of a pungent suffocating smell, and possessing very active chemical properties.

Chlorous; capable of being substituted for chlorine in its combinations with hydrogen or the metals.

Cleavage, Plane of, the plane in which crystals have a tendency to separate.

Cohesion, from *con*, together, and *hæreo*, to stick; the power which causes the molecules of a body to cling together and resist separation.

Combustion, from *comburo*, to burn; the disengagement of light and heat, which frequently accompanies chemical combination.

Conduction, from *con*, together, and *duco*, to lead; the power of transmitting heat or electricity, without change in the relative position of the particles of the conducting body.

Congelation, from *con*, together, and *gelo*, to freeze; the process of freezing.

Constituent, from *constituo*, to put together; that of which anything consists, or is made up.

Contraction, from *con*, together, and *traho*, to draw; the state of being drawn into a narrow compass, or becoming smaller.

Convex, from *con*, together, and *veho*, to carry; curved outwardly or protuberant.

Corpuscular, from *corpus*, a body; composed of, or relating to, atoms.

Crystallography, from *κρύσταλλος*, a crystal or ice, and *γράφω*, to describe; the science which treats of crystals.

Crystallization; the formation of crystals during the passage of certain substances from a fluid to a solid state.

Cube, -ic; a solid figure contained by six equal squares.

Cyanogen, from *κύανος*, blue, and *γεννάω*, to produce; a colourless gas composed of carbon and nitrogen. Its chemical properties much resemble those of chlorine; it derives its name

from the circumstance of its entering into the composition of Prussian-blue.

CYANIDE; a compound of cyanogen with a metal is called a cyanide: as cyanide of potassium.

DECOMPOSITION; the resolution of a compound substance into its component parts.

DECREPITATION, from *de*, from, and *crepito*, to crackle; the crackling noise which certain salts make when heated, usually caused by the sudden escape of steam.

DEFLAGRATION, from *deflagro*, to burn; burning very rapidly.

DELIQUESCENCE, from *deliqueo*, to melt; a gradual melting or dissolving, caused by the absorption of water from the atmosphere.

DENSITY, from *densus*, thick; vicinity or closeness of particles; specific weight.

DEOXIDIZE; to deprive of oxygen.

DETONATION, from *detono*, to thunder; explosion accompanied with noise.

DIALYSIS, from *διὰ*, through, and *λύω*, to loosen; the separation of crystallizable from uncrystallizable substances, by diffusion of their solutions through membranes.

DIAPHANOUS, from *διὰ*, through, and *φαίνω*, to shine; that which allows a passage to the rays of light, but disperses them so as to prevent direct vision.

DIATHERMANOUS, from *διὰ*, through, and *θερμὸς*, heat; that through which heat will pass, is said to be *diathermanous*.

DIMORPHOUS, from *δὶς*, twice, and *μορφὴ*, a form; having two distinct crystalline forms.

DISINTEGRATION, from *dis*, meaning separation, and *integer*, whole; a complete separation of particles.

DISTILLATION, a separation drop by drop; the process by which a fluid is separated from another substance by being first converted into vapour, and afterwards condensed drop by drop.

DODECAHEDRON, from *δώδεκα*, twelve, and *ἕδρα*, a base or side; a solid figure contained by twelve equal sides.

EBULLITION, from *ebullio*, to boil, the act of boiling.

EFFERVESCENCE, the escape of bubbles of gas formed in a liquid, as when marble is decomposed by hydrochloric acid.

EFFLORESCENCE, from *effloresco*, to blow as a flower; the formation of small crystals on the surfaces of bodies in consequence of the abstraction of water from them by the atmosphere.

Electricity, from ἤλεκτρον, amber; the name of a power of matter, which produces a variety of peculiar phenomena, the first of which were observed in the mineral substance called amber; the laws, hypotheses, and experiments by which they are explained and illustrated constitute the science of electricity.

Electrode, from ἤλεκτρον, electricity, and ὁδός, a way; the point at which an electric current enters or quits the body through which it passes.

Electrolysis, -lyte, from ἤλεκτρον, electricity, and λύω, to loosen; the act of decomposing bodies by electricity.

Element, from *elementum*, an element; that which cannot be resolved into two or more parts, and contains but one kind of ponderable matter.

Empyreumatic, from ἐν, in, and πῦρ, fire; having the taste or smell of burnt animal or vegetable substances.

Endosmose, from ἔνδον, within ὠσμος, the act of pushing; a flowing from the outside to the inside.

Equivalent, from *æquus*, equal, and *valeo*, to be worth; equal in value, or in the power of combining with other substances.

Evaporation, from *e*, out, and *vapor*, vapour; the conversion of a liquid into vapour.

Exosmose, from ἔξω, without, and ὠσμος, the act of pushing; a flowing from the inside to the outside.

Expansion, from *expando*, to open out; the enlargement or increase in the bulk of bodies, which is produced by heat.

Experiment, from *experior*, to attempt, to try; something done in order to discover an uncertain or unknown effect.

Explosion, from *ex*, out, and *plaudo*, to utter a sound; a sudden expansion of an elastic fluid, with force and a loud report.

Fermentation, from *fermentum*, that which is light and puffy; originally applied to the process by which alcohol is formed in saccharine liquids. A metamorphosis or transposition of particles caused by contact with a substance in a state of chemical change.

Ferruginous, from *ferrum*, iron; belonging to, or containing iron.

Filter, a strainer.

Fluorine, from *fluo*, to flow; an elementary principle contained in fluor spar, which is so called from its acting as a flux in the working of certain minerals.

Flux, from *fluo*, to flow; that which assists in the fusion of other substances when mixed with it.

FOCUS, from *focus*, a fire-place; a point in which a number of rays of light or heat meet, after being refracted or reflected.

GAS, a permanent aëriform fluid.

GELATINOUS, from *gelo*, to freeze, resembling jelly.

GRAVITY, from *gravis*, heavy; the natural tendency of bodies to fall towards a centre, usually the centre of the earth.

GRAVITY, SPECIFIC; the relative gravity or weight of a body, considered with regard to an equal bulk of some other body, which is assumed as a standard of comparison.

HETEROGENEOUS, from ἕτερος, different, and γένος, kind; different in nature and properties.

HOMOGENEOUS, from ὁμὸς, like, and γένος, kind; alike in nature and properties.

HOMOLOGOUS, from ὁμὸς, like, and λόγος, a relation; a term applied to a series of compounds increasing by like additions of the same elements.

HYDRATE, from ὕδωρ, water; any substance which contains water chemically combined.

HYDROGEN, from ὕδωρ, water, and γεννάω, to produce; an inflammable, colourless, and aëriform fluid; the lightest of all known substances, and one of the elements of water.

HYDRO; when prefixed to the name of a chemical substance, denotes that hydrogen enters into the composition of the substance which it signifies.

HYDROSTATICS, from ὕδωρ, water, and στατὸς, poised; the branch of Natural Philosophy, which treats of the pressure and equilibrium of non-elastic fluids, and also of the weight, pressure, &c., of solids immersed in them.

HYPO, from ὑπὸ, under; when prefixed to a word, denotes an inferior quantity of some ingredient which enters into the composition of the substance which it signifies.

HYPOTHESIS, from ὑπὸ, under, and τίθημι, to place; a principle supposed, or taken for granted, in order to prove a point in question.

IGNITE, from *ignis*, fire; to heat a substance to redness; to set on fire.

IMPONDERABLE, from *in*, not, and *pondero*, to weigh; that which has no perceptible weight.

INCANDESCENT, from *incandesco*, to grow white; white or glowing with heat.

INCREMENT, from *incresco*, to increase; the quantity by which anything increases or becomes greater.

INDUCTION, ELECTRICAL, from *in*, to, and *duco*, to lead; the effect produced by the tendency of an insulated electrified body to excite an opposite electric state in neighbouring bodies.

INERTIA, from *inertia*, inactivity; the disposition of matter to remain in its state of rest or motion.

INFLAMMABLE, from *in*, and *flamma*, a flame; capable of burning with a flame.

INSULATION, from *insula*, an island; when a body containing a quantity of free heat or electricity is surrounded by non-conductors, it is said to be *insulated.*

INTERSTICES, from *interstitium*, a break or interval; the unoccupied spaces between the molecules of bodies.

IODIDE; a compound of iodine and a metal.

IODINE, from ἴον, a violet, and εἶδος, the form or likeness; a soft opaque elementary substance, which, when heated, sublimes in the form of a violet-coloured vapour.

ISOMERIC, from ἴσος, equal, and μέρος, a part; substances which consist of the same ingredients, in the same proportions, and yet differ essentially in their properties, are called *isomeric*.

LAMINÆ, from *lamina*, a thin plate; extremely thin plates, of which some solid bodies are composed.

LEVIGATION, from *lævis*, smooth; the reducing of hard bodies to a very fine powder, by grinding with water.

LIGNIN, from *lignum*, wood; an organic principle found in ligneous structures.

LITMUS; a blue pigment obtained from the lichen *roccella;* it is a most delicate test for acids, which turn it red.

MALLEABLE, from *malleus*, a hammer; that which is capable of being spread out by hammering.

METALLURGY, from μέταλλον, a metal, and ἔργον, a work; the art of working metals, and separating them from their ores.

MOLECULES, -AR, a diminutive from *moles*, a mass; the infinitely small material particles, of which bodies are conceived to be aggregations; the smallest particles capable of independent existence.

MUCILAGINOUS; resembling mucilage or gum.

MUREXIDE, from *murex*, a fish affording a purple dye; a beautiful

purple compound, resulting from the decomposition of uric acid by means of nitric acid.

NASCENT, from *nascor*, to be born; in the moment of formation.

NITROGEN, from *νίτρον*, nitre, and *γεννάω*, to produce; a colourless elementary gas, devoid of taste and smell; it is one of the constituents of the atmosphere, and also of nitric acid, from which latter circumstance it derives its name.

NITROGENOUS; containing nitrogen in combination.

NUCLEUS, from *nucleus*, a kernel; the central parts of a body, which are supposed to be firmer, and separated from the other parts, as the kernel of a nut is from the shell; also, the point about which the matter is collected.

OCTOHEDRON, -AL, from *ὀκτώ*, eight, and *ἕδρα*, a side; a solid figure contained by eight equal and equilateral triangles.

OLEFIANT GAS, from *oleum*, oil, and *fio*, to become; a colourless gas, composed of carbon and hydrogen, which derives its name from its property of forming an oil-like liquid with chlorine.

ORGANIC MATTER, from *ὄργανον*, an organ; matter of which the organic parts or juices of plants and animals are composed, or which is derived from such parts by the action of chemical agents, is called *organic*.

OXIDE; a compound of oxygen with a metal or non-metallic body, not having acid properties.

OXIDIZE; to combine with oxygen.

OXYGEN, from *ὀξὺς*, acid, and *γεννάω*, to produce; a colourless elementary gas, which was formerly supposed to be the universal acidifying principle.

PELLICLE, a diminutive from *pellis*, a skin or crust; a thin crust formed on the surface of a solution.

PERCOLATE, from *per*, through, and *colo*, to strain; to strain through.

PERMEATE, from *permeo*, to pass through; to penetrate.

PHENOMENON, from *φαίνομαι*, to appear; an appearance which is more or less remarkable.

PHILOSOPHY, from *φιλέω*, to love, and *σοφία*, wisdom; the study or knowledge of nature or morality, founded on reason and experience; the word originally implying "A love of wisdom."

PHLOGISTON, from *φλέγω*, to burn; a name given by the older chemists to an imaginary substance, which was considered as the principle of inflammability.

PHOSPHORUS, from φῶς, light, and φέρω, to produce; a highly inflammable elementary substance, obtained from calcined bones, which emits light when placed in the dark, owing to its undergoing a slow combustion.

PHYSICS, -ICAL, from φύσις, nature; the science of natural bodies, their phenomena, causes, and effects, with their affections, motions, and operations.

PNEUMATICS, from πνεῦμα, air; that branch of Natural Philosophy which treats of the weight, elasticity, and other properties of aëriform fluids.

POLARITY; the opposition of two equal forces in bodies, similar to that which confers the tendency of magnetized bodies to point towards the magnetic poles.

POLARIZED LIGHT; light which, by reflection or refraction at a certain angle, or by refraction in certain crystals, has acquired the property of exhibiting opposite effects in planes at right angles to each other, is said to be *polarized.*

PORES, from πόρος, a passage; the small interstices between the solid particles of bodies.

PRECIPITATION, from *præcipito*, to fall suddenly; the formation and separation of a solid substance in a liquid.

PRODUCT, from *pro*, forth, and *duco*, to draw; anything formed from the elements of another by an operation.

PYRO, from πύρ, fire; when prefixed to a word, denotes that the substance which it signifies has been formed at a high temperature.

QUALITATIVE; regarding the properties of a body, and the kinds of matter of which it is composed, without reference to quantity.

QUANTITATIVE; regarding quantities.

RADIATION, from *radius*, a ray; the shooting forth in all directions from a centre.

RADICAL (or *radicle*), an element; or a group of elements capable of being transferred from one form of combination to another without decomposition.

RAREFACTION, from *rarus*, rare, and *facio*, to make; the act of causing a substance to become less dense; it also denominates the state of this lessened density.

RECTIFICATION; the process of drawing anything off by distillation, in order to obtain it in a state of greater purity.

REFRACTION, from *re*, back, and *frango*, to break; the deviation

of rays of light or heat from their direct course, when passing through media of different densities.

Refrigeration, from *re*, again, and *frigus*, cold; the act of cooling.

Repulsion, from *re*, back, and *pello*, to drive; that property in certain bodies, whereby they mutually tend to recede from each other.

Salifiable bases, from *sal*, salt, and *fio*, to become; bodies capable of combining with acids, to form salts.

Sapid, from *sapio*, to taste of; possessing the power of exciting the organs of taste.

Saturation, -ated, from *satur*, full; the solution of one body in another until the receiving body can contain no more. A solution is said to be saturated with an acid or an alkali, when the latter is added in sufficient quantity to render it neutral, and *supersaturated* when the point of neutrality has been exceeded.

Solution, from *solvo*, to loosen or melt; any liquid which contains another substance dissolved in it.

Solvent; any substance which will dissolve another.

Specific, from *species*, a particular sort or kind; that which denominates any property which is not general, but is confined to an individual or species.

Specific gravity; see Gravity, specific.

Spectrum; the image formed by the refraction and dispersion of luminous rays by a prism.

Sublimation, from *sublimis*, high; the act of raising into vapour by means of heat, and condensing in the upper part of the vessel in a solid state.

Sulphide (Sulphuret); a combination of a metal with sulphur.

Supersaturate; see Saturation.

Ternary, from *ter*, thrice; containing three units.

Tetrahedron, from τέσσαρες, four, and ἕδρα, a base or side; a solid figure contained by four equal and equilateral triangles.

Titration; the determination of the strength of a solution by volumetric analysis.

Transparent; a term to denote the quality of a substance which not only admits the passage of light, but also the images of external objects.

Triturate, from *trituro*, to thresh; to reduce to powder.

VACUUM, from *vacuus*, empty; a space empty, and devoid of all matter.

VOLUME, from *volumen*, a roll; the apparent space occupied by a body.

VOLUMETRIC ANALYSIS; analysis by measurement of solutions.

WEIGHT; the pressure which a body exerts vertically downwards in consequence of the action of gravity.

ZERO; the numeral 0, which fills the blank between the ascending and descending numbers of a series.

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THE END.

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